298449

QUALITY ASSURANCE PROJECT PLAN ADDENDUM
REMEDIAL DESIGN/REMEDIAL ACTION
SOUTHEAST ROCKFORD GROUNDWATER CONTAMINATION SITE
ROCKFORD, ILLINOIS

April 20, 2005

by

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NES Project: 1016-2

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Southeast Rockford Groundwater Contamination Site
Quality Assurance Project Plan Addendum

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Southeast Rockford Groundwater Contamination Site
RD/RA QAPP Addendum Summary

The Quality Assurance Project Plan (QAPP) was prepared for the Ground Water Response Action at the Southeast Rockford Groundwater Contamination Site (Site) by Nationwide Environmental Services Inc., (NES) for the City of Rockford (City) and submitted in September 1998 as a deliverable in accordance with the US Environmental Protection Agency (USEPA) Statement of Work (SOW) and Consent Decree (CD). The Site QAPP was prepared by NES in accordance with USEPA QAPP guidance documents, in particular, the Contract Laboratory Program (CLP) guidelines (USEPA, 1986d), Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans (USEPA, 1986b), the Region V Model QAPP (USEPA, 1991c) and the EPA Requirements for Quality Assurance Project Plans For Environmental Data Operations, (USEPA, 1993).

The CD issued for the Site required that environmental monitoring and measurement efforts mandated or supported by USEPA participate in a centrally managed quality assurance (QA) program. Any party generating data under this program has the responsibility to implement minimum procedures to assure that the precision, accuracy, representativeness, completeness, and comparability (PARCC) of its data are known and documented. To ensure this responsibility is met uniformly, each party is to prepare a written QAPP covering each project it is to perform.

The QAPP submitted to USEPA to meet Site monitoring and analytical requirements of the selected remedy presented the organization, objectives, functional activities, and specific quality assurance (QA) and quality control (QC) activities associated with the performance of the Remedial Design and Remedial Action (RD/RA) at the Site. The QAPP also described the specific protocols to be followed for sampling, sample handling and storage, chain of custody, and laboratory analysis. The QAPP is a companion document to the RD/RA Work Plan prepared for the groundwater monitoring network construction component of the Site remedy and addressed the Site remedial design, construction and operation and maintenance phases to be performed to complete the remedial actions contained in the RD/RA Work Plan. The Field Sampling Plan (FSP), and the Health and Safety Plan (HASP), are also companion documents to the RD/RA

Work Plan submitted to the USEPA during the start-up phase of the remedy construction in September 1998, and together with the RD/RA Work Plan are intended to guide personnel in the conduct and reporting of activities associated with the performance of the RD/RA Work Plan.

The remedy design and construction phases of the ground water monitoring network component of the remedy were completed in September, 1999 and long-term operation and maintenance (O&M) is now occurring. Remedy construction completion was documented in the Remedial Action Report issued in September 1999. A Five Year Review was conducted for the Site in May 2003 by USEPA and the Site was determined to remain operational and functional and protective of human health and the environment. Current O&M activities for the ground water monitoring network principally involve collection and analysis of samples from established groundwater monitoring locations, and maintaining the well heads to preserve access to the wells

The procedures identified in the RD/RA QAPP have been implemented in accordance with applicable professional technical standards, USEPA requirements, government regulations and guidelines, and specific project goals and requirements since inception of the Site RD/RA project construction activities in 1998. However, certain parties identified in the original QAPP and contractor entities have changed during the term of the remedy. This addendum to the RD/RA QAPP is submitted to revise the personnel contact information identified in the original QAPP and update the quality assurance reference documents provided as Appendix A to the QAPP. The updated quality assurance reference documents include:

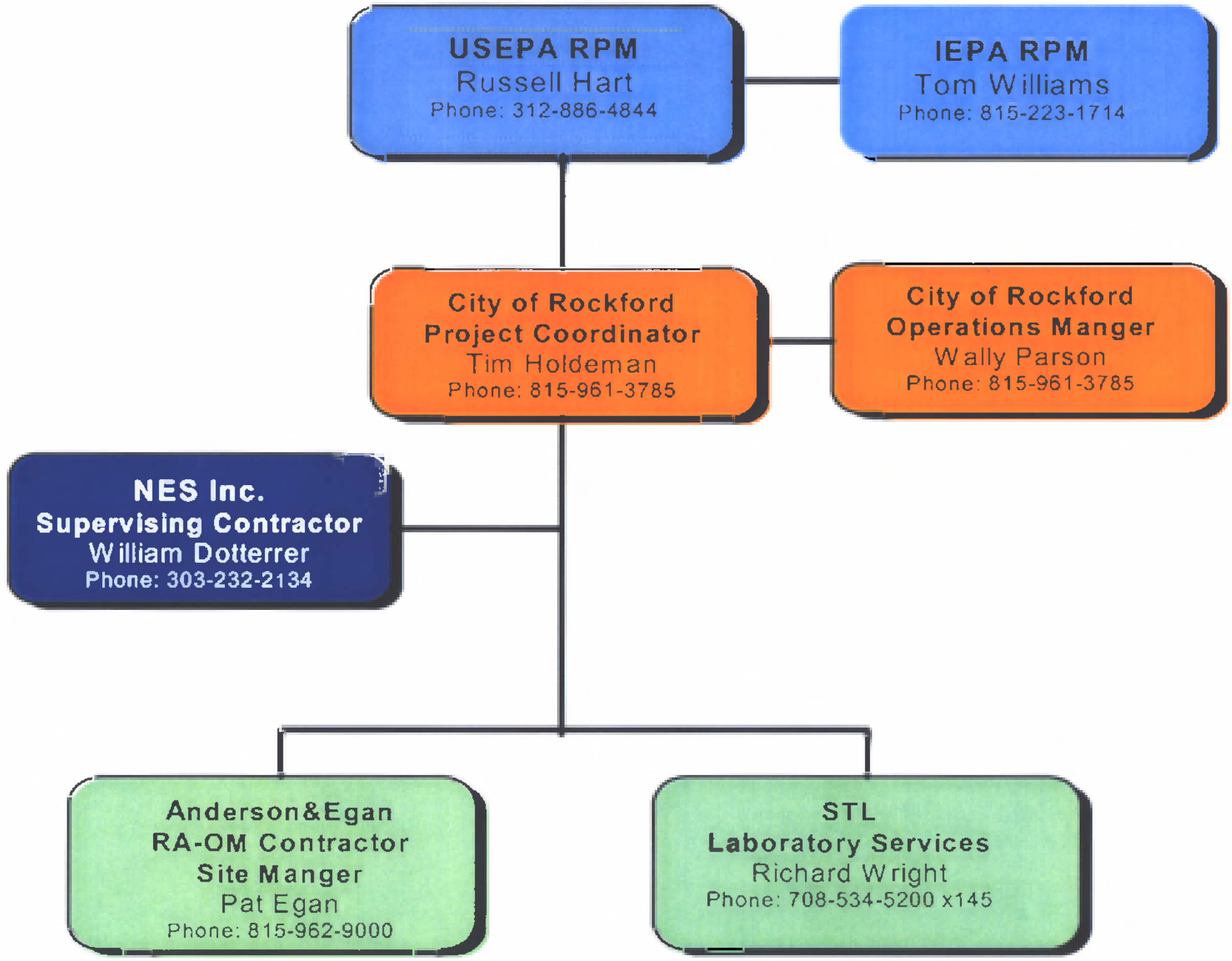
- Laboratory Quality Manual; and
- Laboratory Standard Operating Procedure

The responsibilities, of the key project personnel as provided in the RD/RA QAPP are summarized for each participating party below. An updated project organizational chart is provided in Figure 1.

Party	Position	Responsibilities
City of Rockford	Project Coordinator Tim Holdeman	Representative responsible for actions required in the CD, ROD and RD/RA SOW
	Project Manger Wally Parson	Representative responsible for coordinating field activities with Supervising Contractor
Nationwide Environmental Services	Supervising ContractorWilliam Dotterrer	Representative responsible for coordinating actions in the RD/RA Work Plan with duties to include: • Project administration • Overall technical direction of the project • Supervision of project teams • Primary liaison among the City, Subcontractors, USEPA, and IEPA • Coordination, preparation, and approval of all project deliverables • Preparation for and attendance at project meetings
Anderson&Egan	Project Manager Pat Egan	Representative responsible for overseeing groundwater sample collection and transfer to laboratory support subcontractor, and maintenance of groundwater monitoring network • Coordinating field activities • Supervision of field crews • Filed log books • Chain of custody • Data validation • Data completeness
Severn Trent Laboratories	Project Coordinator Richard Wright	Representative responsible for coordinating laboratory services to support project to include: • Liaison with NES and STL technical staff • Monitor workloads and ensure availability of resources • Analytical report preparation and overview • In-house chain-of-custody supervision • Quality assurance oversight

RD/RA Project
Organization Chart

RD/RA PROJECT
ORGANIZATION CHART



Severn Trent Laboratories, Inc.
Laboratory Quality Manual

SEVERN TRENT STL

LABORATORY QUALITY MANUAL

STL Chicago
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Approved by (Signature / Date):

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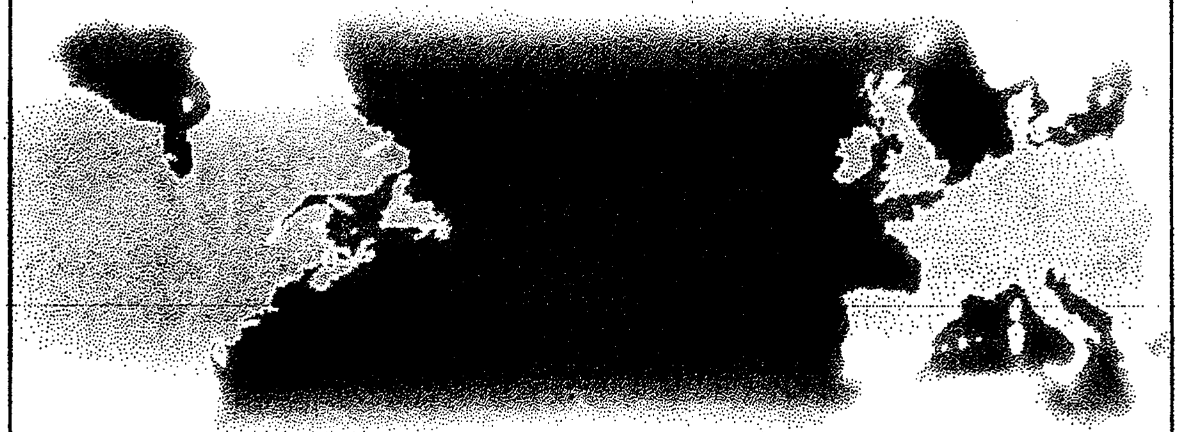
SEVERN TRENT STL

Vision

STL will be the recognized industry leader for environmental analysis.



Mission

Through the innovation and dedication of our people, together with the quality of our systems, we will deliver levels of performance that delight our clients, retain the confidence of our stakeholders and enable the profitable growth of our business.

Severn Trent Laboratories

1.0 Introduction, Purpose, and Scope

1.1 STL Overview

STL Chicago (STL) is a part of Severn Trent Laboratories, a major group of U.S. based companies. The companies are owned by Severn Trent, plc, an international provider of water and wastewater services headquartered in Birmingham, UK.

STL is a full-service environmental laboratory that provides quality comprehensive and integrated professional analytical services effectively and efficiently. A broad range of environmental testing services are offered that span a variety of matrices including aqueous, saline, solid, tissue and drinking water.

Associated with this activity are services to assure client requirements are known, communicated and satisfactorily addressed, and a deliverables package presenting the analytical results. The laboratory provides expert personnel for supervision, technical consultation, and project review for effective planning and implementation of analytical assignments.

STL operates under the regulations and guidelines of the following federal programs:

- Air Force Center for Environmental Excellence (AFCEE)
- US Army Corp of Engineers, Hazardous, Toxic and Radioactive Waste (USACE HTRW)
- Clean Water Act (CWA)
- Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)
- Navy Facilities Engineering Service Center (NFESC)
- National Pollution, Discharge, and Elimination System (NPDES)
- Occupational Safety and Health Administration (OSHA)
- Resource Conservation and Recovery Act (RCRA)
- Safe Drinking Water Act (SDWA)
- Toxic Substances Control Act (TSCA)

STL also provides services under various state and local municipal guidelines. A current table of analytical services, list of certifications and general service listing is presented on the MySTL webpage or available from the laboratory. www.stl-inc.com

1.2 Quality Assurance Policy

It is STL's policy to:

- Provide high quality, consistent, and objective environmental testing services that meet all federal, state, and municipal regulatory requirements.
- Generate data that are scientifically sound, legally defensible, meet project objectives, and are appropriate for their intended use.
- Provide STL clients with the highest level of professionalism and the best service practices in the industry.
- Build continuous improvement mechanisms into all laboratory, administrative, and managerial activities.
- Maintain a working environment that fosters open communication with both clients and staff and ensures data integrity.

1.3 Management Commitment to Quality Assurance

STL management is committed to providing the highest quality data and the best service in the environmental testing industry. To ensure that the data produced and reported by STL meet the requirements of its clients and comply with the letter and spirit of municipal, state and federal regulations, STL maintains a quality system that is clear, effective, well communicated, and supported at all levels in the company.

Line organizations verify that specifications are achieved; QA organizations assist and provide oversight and verification of processes through planning, reviews, audits, and surveillances. The quality objectives are derived from this Laboratory Quality Manual (LQM), Standard Operating Procedures (SOPs) and Work Instructions.

1.4 Purpose

The purpose of the LQM is to describe STL's Quality System and to outline how that system enables all employees to meet the Quality Assurance (QA) policy. This LQM also describes specific QA activities and requirements and prescribes their frequencies. Roles and responsibilities of management and laboratory staff in support of the Quality System are also defined in this LQM.

1.5 Scope

This LQM is specific to STL Chicago's quality systems and laboratory operation's. All other STL locations have LQMs under the Corporate Quality Management Plan (QMP) or the Corporate QMP itself.

The laboratory is committed to ensuring that resources are available and deployed to meet client expectations. This includes gathering project information prior to sample receipt to ensure client expectations will be met with respect to:

- Sampling containers;
- Analytical methods employed;

- Accuracy and precision;
- Reporting limits;
- Personnel qualifications, training, and experience;
- Calibration and quality control measures employed;
- Regulatory requirements;
- Report contents;
- Supporting documentation, records and evidence; and
- Review of data

1.6 Servicing

Project Managers are the direct client contact and they ensure resources are available to meet project requirements. Although Project Managers do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure that available resources are sufficient to perform work for the client's project. Project Managers provide a link between the client and laboratory resources.

The laboratory has established procedures for performing and verifying that client servicing meets requirements. Typical services provided are:

- Sample Containers/Supplies – *Container Management: Process Operation* (UCM-001)
- Project QAP preparation – *Project Planning Process* (UPM-003)
- Regulatory advisory functions – *Project Planning Process* (UPM-003)
- Consulting – *Project Planning Process* (UPM-003)

Regulatory and advisory functions are addressed under the same procedures used for project planning.

2.0 References

The following references were used in preparation of this document and as the basis of the STL Quality System:

EPA Guidance for Preparing Standard Operating Procedures (SOPs), EPA QA/G-6, US EPA, Office of Environmental Information, EPA/240/B-01/004, March 2001.

EPA Requirements for Quality Management Plans, EPA QA/R-2, US EPA, Office of Environmental Information, EPA/240,B-01/002 March 2001.

EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, US EPA, Office of Environmental Information, EPA/240/B-01/003, March 2001.

EPA Quality Manual for Environmental Programs, 5360 A1, US EPA Office of Environmental Information – Quality Staff, May 2000.

General Requirements for the Competence of Testing and Calibration Laboratories, ISO/IEC 17025, December 1999.

Good Automated Laboratory Practices, Principles and Guidance to Regulations for Ensuring Data Integrity in Automated Laboratory Operations with Implementation Guidance, EPA 2185, US EPA Office of Information Resources Management, August 1995.

Air Force Center for Environmental Excellence (AFCEE) Quality Assurance Project Plan (QAPP), Version 3.1, August 2001.

National Environmental Laboratory Accreditation Conference, Constitution, Bylaws, and Standards, EPA 600/R-00/084, US EPA Office of Research and Development, June 2000.

Navy Installation Restoration Laboratory Quality Assurance Guide, Interim Guidance Document, Naval Facilities Engineering Service Center (NFESC), February 1996.

Navy Installation Restoration Chemical Data Quality Manual, Navy IR CDQM, Special Publication SP-2056-ENV, September 1999.

Department of Defense Quality Systems Manual for Environmental Laboratories, Version 1, October 2000.

Shell for Analytical Chemistry Requirements, US Army Corps of Engineers, EM 200-1-3, Appendix I, February 2001

This LQM was written to comply with the National Environmental Laboratory Accreditation Conference (NELAC) standards. Refer to Table 1 for a cross-section comparison of this LQM to the NELAC standards.

Table 1.

Correlation of QAPP Sections with NELAC 5.5.2 Quality Manual Requirements

NELAC Chapter 5.5.2 Quality Manual	Laboratory Quality Manual Section
a. Quality policy statement, including objectives and commitments	1.2 Quality Assurance Policy 4.2.1 Objectives of the Quality System
b. Organization and management structure	4.1 Organization and Management
c. Relationship between management, technical operations, support services and the quality systems	4.1.2 Roles and Requirements 4.2 Quality System
d. Records retention procedures; document control procedures	4.3 Document Control 4.12.2 Record Retention
e. Job descriptions of key staff and references to job descriptions of other staff	4.1.2 Roles and Requirements
f. Identification of laboratory approved signatories	4.1 Organization and Management
g. Procedures for achieving traceability of measurements	5.5 Measurement Traceability
h. List of all test methods under which the laboratory performs its accredited testing	5.3.1 Method Selection
i. Mechanisms for assuring the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work	4.4.2 Project-Specific Quality Planning

Table 1.

Correlation of QAPP Sections with NELAC 5.5.2 Quality Manual Requirements

NELAC Chapter 5.5.2 Quality Manual	Laboratory Quality Manual Section
j. Reference to the calibration and/or verification test procedures used	5.3.4 Method Verification 5.3.5 Method Validation & Verification Activities 5.3.6 Data Reduction & Review 5.4.3 Equipment Verification and Calibration
k. Procedures for handling submitted samples	4.7.1 Sample Acceptance Policy 5.7 Sample Handling, Transport and Storage
l. Reference to the major equipment and reference measurement standards used as well as the facilities and services used in conducting tests	1.6 Servicing 4.1.1 Laboratory Facilities 4.6 Purchasing Services & Supplies 5.2 Facilities 5.4.2 Equipment Maintenance 5.4.3 Equipment Verification and Calibration
m. Reference to procedures for calibration, verification and maintenance of equipment	5.4.2 Equipment Maintenance 5.4.3 Equipment Verification and Calibration
n. Reference to verification practices including inter-laboratory comparisons, proficiency testing programs, use of reference materials and internal QC schemes	5.8.1 Proficiency Testing 5.8.2 Control Samples
o. Procedures for feedback and corrective action whenever testing discrepancies are detected, or departures from documented procedures occur	4.8 Complaints 4.9 Control of Non-Conformances 4.10 Corrective Action 4.11 Preventive Action 5.8.6 Permitting Departures from Documented Procedures
p. Laboratory management arrangements for exceptionally permitting departures from documented policies and procedures	4.4.1 Contract Review 4.4.2 Project-Specific Quality Planning 5.8.6 Permitting Departures from Documented Procedures
q. Procedures for dealing with complaints	4.8 Complaints
r. Procedures for protecting confidentiality and proprietary rights	4.7.2 Client Confidentiality and Proprietary Rights
s. Procedures for audits and data review	4.13 Internal Audits 4.14 External Audits 5.3.6 Data Reduction and Review
t. Process/procedures for establishing that personnel are adequately experienced in duties they are expected to carry out and are receiving any needed training	5.1.2 Training
u. Ethics policy statement developed by the laboratory and training personnel in their ethical & legal responsibilities	5.1.3 Ethics Policy
v. Reference to procedures for reporting analytical results	5.3 Test Methods 5.3.6 Data Reduction and Review 5.9 Project Reports
w. Table of contents, listing reference, glossaries and appendices	TOC Table of Contents Appendix List of Cited SOPs and Work Instructions

3.0 Terms and Definitions

Accuracy: The degree of agreement between a measurement and true or expected value, or between the average of a number of measurements and the true or expected value.

Audit: A systematic evaluation to determine the conformance to specifications of an operational function or activity.

Batch: Environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of 1 to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (e.g., volatile organics, water), the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Chain of Custody (COC): A system of documentation demonstrating the physical possession and traceability of samples.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): Legislation (42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and reauthorization Act of 1986 (SARA), 42 U.S.C. 9601et seq.

Compromised Sample: A sample received in a condition that jeopardizes the integrity of the results. See Section 4.7.1 for a description of these conditions.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products.

Confirmation: Verification of the presence of a component using an additional analytical technique. These may include second column confirmation, alternate wavelength, derivatization, mass spectral interpretation, alternative detectors, or additional cleanup procedures.

Corrective Action: Action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

Demonstration of Capability (DOC): Procedure to establish the ability to generate acceptable accuracy and precision.

Detection Limit Check Standard (DLCK): A non-processed standard spiked at approximately ½ the method reporting limit. Used in conjunction with the MRL Check standard in LGC analysis.

Equipment Blank (EB): A portion of the final rinse water used after decontamination of field equipment; also referred to as Rinsate Blank and Equipment Rinsate.

Extraction Blank (EB1, EB2, EB3): A blank that has been taken through the extraction procedure such as TCLP/SPLP; 5035, AVS/SEM.

Document Control: The act of ensuring that documents (electronic or hardcopy and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): Legislation under 7 U.S.C. 135 et seq., as amended.

Federal Water Pollution Control Act (Clean Water Act, CWA): Legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat. 816.

Field Blank (FB): A blank matrix brought to the field and exposed to field environmental conditions.

Field Duplicate (FD): Duplicate field-collected sample.

Field of Testing (FOT): A field of testing is based on NELAC's categorization of accreditation based on program, matrix and analyte.

Good Laboratory Practices (GLP): Formal regulations for performing basic laboratory operations outlined in 40 CFR Part 160 and 40 CFR Part 729 and required for activities performed under FIFRA and TSCA.

Holding Time: The maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Instrument Blank: A blank matrix that is the same as the processed sample matrix (e.g. extract, digestate, condensate) and introduced onto the instrument for analysis.

Internal Chain of Custody (COC): An unbroken trail of accountability that ensures the physical security of samples, data and records. Internal COC refers to additional documentation procedures implemented within the laboratory that includes special sample storage requirements, and documentation of all signatures and/or initials, dates, and times of personnel handling specific samples or sample aliquots.

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ±100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (LCS): A blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Laboratory Quality Manual (LQM): A document stating the quality policy, quality system and quality practices of the laboratory. The LQM may include by reference other documentation relating to the laboratory's quality system.

Limit of Detection (LOD): The minimum amount of a substance that an analytical process can reliably detect.

Matrix: The substrate of a test sample. Common matrix descriptions are defined in Table 2.

Table 2. Matrix Descriptions

Matrix	Description
Aqueous	Aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine source. Includes surface water, groundwater, effluents, leachates and wastewaters.
Drinking Water	Aqueous sample that has been designated a potable water source.
Saline	Aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake.
Liquid	Liquid with <15% settleable solids.
Solid	Soil, sediment, sludge, ash, paint chips, filters, wipes or other matrices with ≥15% settleable solids.
Waste	A product or by-product of an industrial process that results in a matrix not previously defined (i.e., drum liquid or oils).
Tissue	Sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Matrix Duplicate (MD): Duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

Matrix Spike (MS): Field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): A replicate matrix spike.

Method Blank (MB): A blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Method Detection Limit (MDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range.

Method Detection Limit Check (MDLCK): A standard that is processed with the MDL Study that is spiked at approximately ½ the low standard or reporting limit in the method.

Method Reporting Limit Check (MRL): A standard that is not processed, is spiked at approximately 2x the low standard or reporting limit. This standard check is used in conjunction with the LCG analysis.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Precision: An estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions.

Preservation: Refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical and/or biological integrity of the sample.

Proficiency Testing: Determination of the laboratory calibration or testing performance by means of inter-laboratory comparisons.

Proficiency Test (PT) Sample: A sample, the composition of which is unknown to the analyst, that is provided to test whether the analyst/laboratory can produce analytical results within specified performance limits. Also referred to as Performance Evaluation (PE) Sample.

Proprietary: Belonging to a private person or company.

Quality Assurance (QA): An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Assurance (Project) Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.

Quality Control (QC): The overall system of technical activities, the purpose of which is to measure and control the quality of a product or service.

Quality Control (QC) Sample: A control sample, generated at the laboratory or in the field, or obtained from an independent source, used to monitor a specific element in the sampling and/or testing process.

Quality Management Plan (QMP): A formal document describing the management policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an agency, organization or laboratory to ensure the quality of its product and the utility of the product to its users.

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA/QC.

Quantitation Limit (QL): The minimum amount of a substance that can be quantitatively measured with a specified degree of confidence and within the accuracy and precision guidelines of a specific measurement system. The QL can be based on the MDL, and is generally calculated as 3-5 times the MDL, however, there are analytical techniques and methods where this relationship is not applicable. Also referred to as Practical Quantitation Level (PQL), Estimated Quantitation Level (EQL), Limit of Quantitation (LOQ).

Raw Data: Any original information from a measurement activity or study recorded in laboratory notebooks, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic/optical media, including dictated observations, and recorded data from automated instruments. Reports specifying inclusion of "raw data" do not need all of the above included, but sufficient information to create the reported data.

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Standard: A standard, generally of the highest metrological quality, available at a given location from which measurements made at that location are derived.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Resource Conservation and Recovery Act (RCRA): Legislation under 42 U.S.C. 321 et seq. (1976).

Safe Drinking Water Act (SDWA): Legislation under 42 U.S.C. 300f et seq. (1974), Public Law 93-523.

Sampling and Analysis Plan (SAP): A formal document describing the detailed sampling and analysis procedures for a specific project.

Selectivity: The capability of a measurement system to respond to a target substance or constituent.

Sensitivity: The difference in the amount or concentration of a substance that corresponds to the smallest difference in a response in a measurement system using a certain probability level.

Spike: A known amount of an analyte added to a blank, sample or sub-sample.

Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

Storage Blank: A blank matrix stored (2-weeks) with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination. OR A blank matrix stored with field samples of a similar matrix.

Systems Audit: A thorough, systematic, on-site, qualitative review of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system.

Test Method: Defined technical procedure for performing a test.

Toxic Substances Control Act (TSCA): Legislation under 15 U.S.C. 2601 et seq., (1976).

Traceability: The property of a result of a measurement that can be related to appropriate international or national standards through an unbroken chain of comparisons.

Trip Blank (TB): A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Verification: Confirmation by examination and provision of evidence against specified requirements.

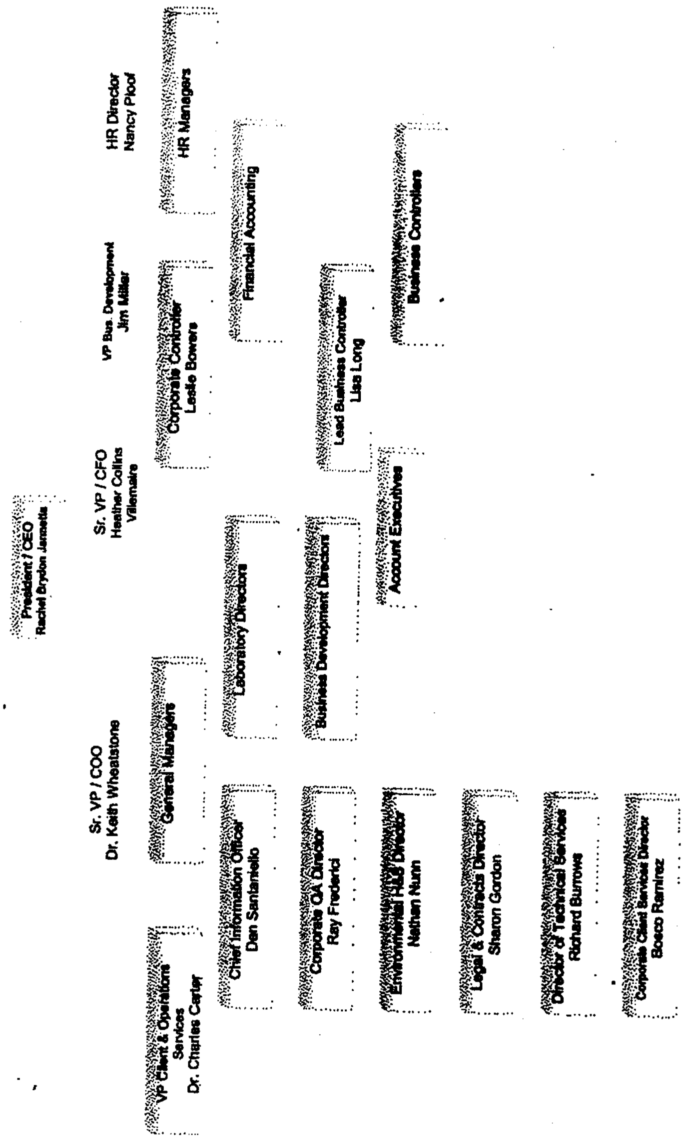
4.0 Management Requirements

The organizational chart of STL is presented in Figure 1. Corporate employees are located at various STL facilities as outlined in the organizational structure. The organizational chart of STL Chicago is presented in Figure 2.

4.1 Organization and Management

The Laboratory Director and Quality Assurance Manager are responsible and have the signature authority for approving and implementing this plan. Additional signatory authorities for the approval of work and release of reports are defined in the *Signature Authority* SOP (UQA-030).

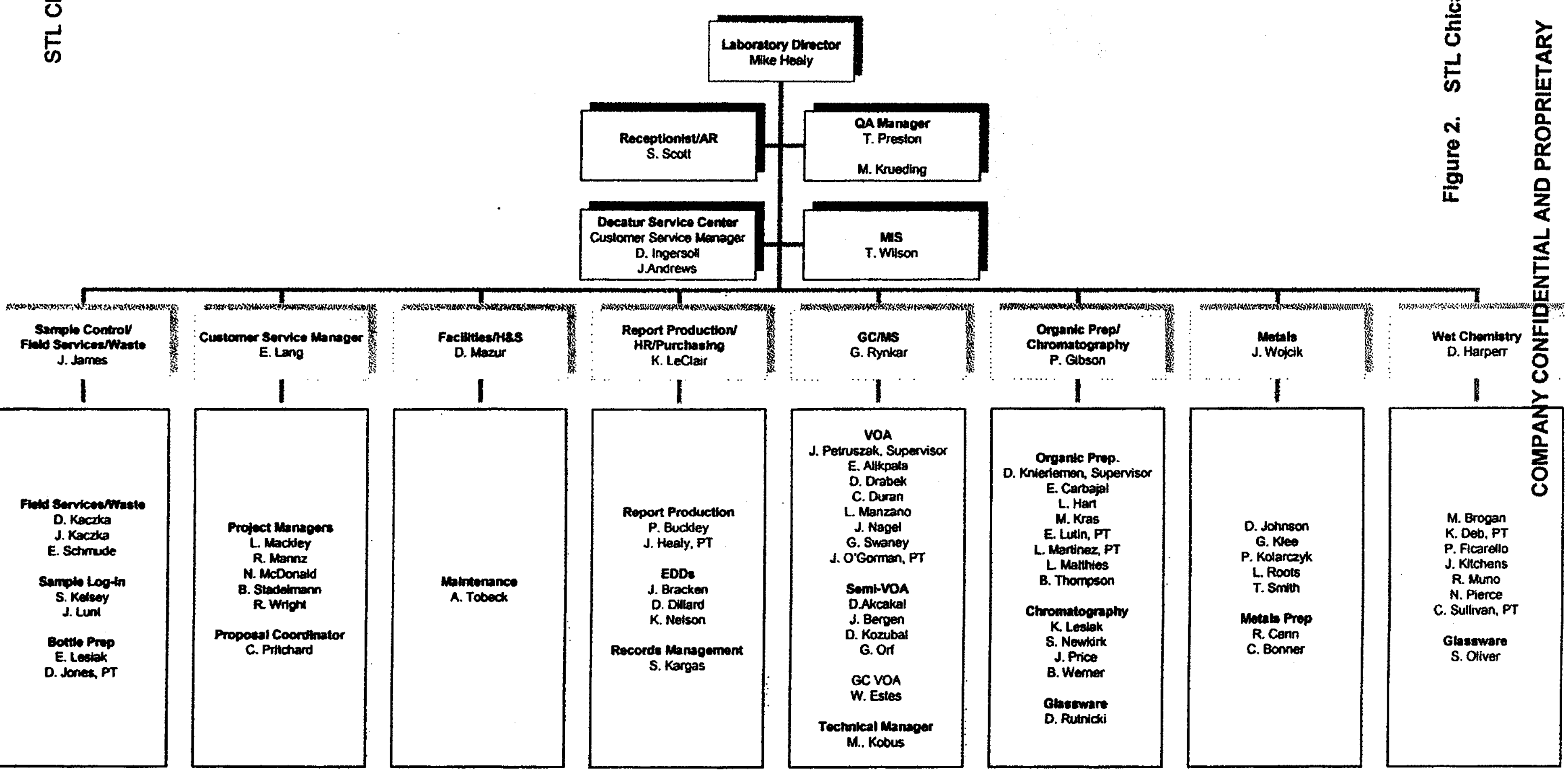
Severn Trent Laboratories, Inc.
Management Structure
January 1, 2004



SEVERN TRENT STL

Figure 1. STL Organization Chart

STL Chicago Operations



May 27, 2004

Figure 2. STL Chicago Organizational Chart

4.1.1 Laboratory Facilities

The laboratory is located in University Park, IL, which is approximately 30 miles south of Chicago, and is staffed by 84 professionals. The laboratory is comprised of 51,000 square feet of state-of-the-art commercial laboratory and office space and houses both inorganic and organic operations. The facility is divided into separate work areas to facilitate sample throughput. These areas include the following:

- Sample receipt and refrigerated storage
- Organic sample preparation
- Glassware preparation
- Metals digestion
- Wet chemistry laboratory
- Instrumentation laboratories

The main instrumentation laboratory is equipped with state-of-the-art instrumentation and sufficient duplicate equipment to provide back-up service for most major systems. A listing of laboratory equipment and instrumentation is referenced as Work Instruction No. CHI-22-09-103. Table 3 is a summary of the major laboratory instruments.

Table 3. Major Equipment List

GC	GC/MS	AA	ICP	CVAA	HPLC	AutoAnalyzer	IC	TOC	TOX
15	14	3	3	2	6	2	2	2	2

Each of these areas has separate heating, ventilation, and air conditioning systems. Non-destructive gas chromatographic detectors and GC/MS rotary pumps are vented out of the instrumentation through charcoal filters.

4.1.2 Roles and Responsibilities

The specific duties and responsibilities of the Laboratory Director, Quality Assurance Manager, Project Managers, Technical Managers, Sample Management Coordination, Data Management Section Manager, Quality Assurance Specialist, Health and Safety Coordinator/Waste Management, Information Technology Manager, and Chemists/Technicians are as follows.

In the absence of any one individual, the staff or assistant within each department is professionally skilled in the ability to administer the function of the administrator or support personnel. This will allow for the continuance of the day-to-day operations of the laboratory.

4.1.2.1 Laboratory Director

The ultimate responsibility for the generation of reliable laboratory data rests with the Laboratory Director, who is accountable to his General Manager and oversees the daily operations of the laboratory. The Laboratory Director's responsibilities include allocation of personnel and resources, setting goals and objectives for both the business and employees, achieving the financial, business and quality objectives of STL. Furthermore, to see that all tasks performed in the laboratory are conducted according to the requirements of this LQM, the Project Technical Profile and/or the appropriate QAPP; and to assure that the quality of service provided complies with the project's requirements.

The Laboratory Director has the authority to affect those policies and procedures to ensure that only data of the highest level of excellence are produced. As such, the Laboratory Director supports a QA Section which has responsibilities independent from sampling and analysis.

The Laboratory Director, with the assistance of the Quality Assurance Manager, has the overall responsibility for establishing policies that ensure the quality of analytical services meet our clients expectations. These policies are defined in this LQM.

4.1.2.2 Quality Assurance Manager

The Quality Assurance (QA) Manager has the full-time responsibility to evaluate the adherence to policies and to assure that systems are in place to produce the level of quality defined in this LQM. The QA Manager is responsible for the approval of IDL/MDL studies, method validation studies, IDOC and CDOC evaluations, the annual review of statistical control limits, data package inspections, and LIMS system method development, validation and maintenance. In addition, the QA Manager assists in the preparation, compilation, and submittal of quality assurance plans; reviews program plans for consistency with organizational and contractual requirements and advises appropriate personnel of deficiencies. The QA Manager is assisted by the QA Specialist in the maintenance of QA records, certifications, accreditations, internal and external audits, corrective action procedures, management of the laboratory's PT Program, and maintenance of training documentation.

The QA Manager shall have the final authority to accept or reject data, and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data. The QA Manager is available to any employee at the facility to resolve data quality or ethical issues. The QA Manager must address any data integrity issue identified internally or externally, establish a corrective action plan and resolve the issue to the client's satisfaction. Issues that involve data recall must be discussed with the Corporate Quality Director Ray Frederici. The QA Manager shall be independent of laboratory operations and has an indirect reporting relationship to the QA Director.

4.1.2.3 Project Managers

The laboratory recognizes the importance of efficient project management. The laboratory Project Managers (PM) are responsible for preparing the Project Technical Profile which summarizes QA/QC requirements for the project, maintaining the laboratory schedule, ensuring that technical requirements are understood by the laboratory, and advising the Laboratory, QA and Technical Managers of all variances. The laboratory Project Manager will provide technical guidance and the necessary laboratory-related information to the preparer of project-specific QAPPs and provide peer review of the final document to ensure accuracy of the laboratory information.

4.1.2.4 Technical Managers

The Technical Managers are the Laboratory Director, laboratory Section Managers and the QA Manager. They are as follows:

- Michael J. Healy, Laboratory Director, BS Environmental Biology,
- 22 years laboratory experience.
- Terese A. Preston, Quality Assurance Manager, BA Biology,
- 20 years laboratory experience.
- Diane L. Harper, Inorganics Section Manager, MA Biology,
- 24 years laboratory experience.
- Jodi L. Wojcik, Metals Section Manager, BS Biology,
- 18 years laboratory experience.
- Patti J. Gibson, Chromatography/Organic Extractions Section Manager, BS Biology,
- 15 years laboratory experience.
- Gary L. Rynkar, GC/MS Section Manager, BS Environmental Biology,
- 16 years laboratory experience.

All of these managers report to the Laboratory Director and serve as the technical experts on assigned projects, provide technical liaison, assist in resolving any technical issues within the area of their expertise; and implement established policies and procedures to assist the Laboratory Director in achieving section goals. The Technical Managers are responsible for ensuring that their personnel are adequately trained to perform analyses; that equipment and instrumentation under their control is calibrated and functioning properly; that system and performance audits are performed on an as-needed basis; provide input and review in the development and implementation of project-specific QA/QC requirements; and for providing the critical review of proposal and project work for programs as directed by the Laboratory Director. The Technical Managers coordinate these activities with the project management and quality assurance sections.

4.1.2.5 Sample Management Coordination

The Project Manager is designated as the Sample Management Coordination for any work subcontracted under their management. The Project Manager verifies each subcontracting request to ensure that special client restrictions are not jeopardized (e.g., samples must be analyzed by the receiving affiliated or network laboratory and must maintain specific certification(s)). The Project Manager is also responsible for verifying the credentials; establishing the service agreement; ensuring data review; and invoicing of all laboratory subcontractors. The Project Manager discusses any deficiencies or anomalies with the subcontractor prior to reporting any data to the client.

4.1.2.6 Data Management Section Manager

The Data Management Section Manager is responsible for coordinating receipt of all data from the various service groups within the laboratory, reviewing data for compliance to laboratory QC criteria and/or criteria in the Project Technical Profile, and ensuring that data are reported in a timely manner and in the proper format.

4.1.2.7 Quality Assurance Specialist

The QA Specialist is responsible for QA documentation and involvement in the following activities:

- Assist the QA Manager in performing the annual internal laboratory audits, compiling the evaluation, and coordinating the development of an action plan to address any deficiency identified.
- Facilitate external audits, coordinating with the QA Manager and Laboratory Staff to address any deficiencies noted at the time of the audit and subsequently presented in the final audit report.
- Assist the QA Manager in the preparation of new SOP's and in the maintenance of existing SOPs, coordinating annual reviews and updates.
- Manages the performance testing (PT) studies, coordinates follow up studies for failed analytes and works with QA Manager and Laboratory Staff to complete needed corrective action reports.
- Personnel training records review and maintenance.
- Document control maintenance.
- Assists the Quality Manager and Project Management Group in the review of program plans for consistency with organizational and contractual requirements. Summarize and convey to appropriate personnel anomalies or inconsistencies observed in the review process.
- Manages certifications and accreditations.
- Monitors for compliance the following QA Metrics: Temperature Monitoring of refrigeration units and incubators; thermometer calibrations; balance calibrations; eppendorf/pipette calibrations; and proper standard/reagent storage.
- Periodic checks on the proper use and review of instrument logs.
- Initiate the Mint-miner data file review process for organic instrumentation. Maintain tracking sheet of activity.
- Initiate the annual Instrument review.
- Assist in the technical review of data packages which require QA review.

4.1.2.8 Health and Safety Coordinator / Waste Management

The Health and Safety Coordinator is responsible for the safety and well-being of all employees while at the laboratory. This includes, but is not limited to, administering the Corporate Safety Manual that complies with federal regulations, MSDS training and review, conducting laboratory safety orientation and tours for all new employees, providing instructions on safety equipment, cleaning up laboratory spills, and instructing personnel of laboratory procedures for emergency situations. The Health and Safety Coordinator is on-call 24-hours a day, 7-days a week for all laboratory situations.

The Health and Safety Coordinator responsibilities additionally include waste management of laboratory generated hazardous waste in accordance with appropriate regulations. This includes maintenance of required documentation, such as waste manifests, segregation of waste in accordance with requirements, and training of personnel in proper segregation of waste.

4.1.2.9 Information Technology Manager

The overall role of the Information Technology (IT) Manager is to enhance laboratory productivity through improved information access, flow, and security. For information to be of greatest value, it must be readily accessible and reliable. It is the responsibility of the IT Manager to provide software tools that allow quick and user friendly access to that information, while at the same time controlling access to that information to those that have the need and proper authority.

Information flow can be enhanced through automation. Automation is the minimization of human intervention in a process. Reduction in human intervention can result in significant error reductions and time savings. The IT Manager assists the laboratory in automation by providing hardware and software solutions to help minimize human intervention in data collection, processing, and storage.

The IT Manager is responsible for providing data security by controlling access, as mentioned above, and for providing for disaster recovery. Data stored on the central Laboratory Information Management System (LIMS, a.k.a., LabNet) is the direct responsibility of the IT Manager. No fewer than two copies of all data should exist at any time so that lost or destroyed data can always be retrieved from an alternate source. These copies may consist of data within the system and on magnetic tape in the case of live data, or two copies on magnetic tape for archived data. Data stored electronically in other departments is the direct responsibility of those departments. However, the IT Manager is responsible for providing procedures and training to all laboratory operations, as appropriate, to assist in making backup copies of local data within the respective operating unit.

STL has established procedures for IT management:

- *Internet Use Policy* – P-I-001
- *Electronic Mail Use* – P-I-002
- *Computer Systems Account and Naming Policy* – P-I-003
- *Computer Systems Password Policy* – P-I-004
- *Software Licensing Policy* – P-I-005
- *Virus Protection Policy* – P-I-006

4.1.2.10 Chemists / Technicians

Any effective laboratory quality assurance/quality control program depends on the entire organization, including management and every individual on the laboratory staff. The initial review for acceptability of analytical results rests with the analysts conducting the various tests. Observations made during the performance of an analytical method may indicate that the analytical system is not in control. Analysts must use quality control indicators to assure that the method is in-control before reporting results.

4.2 Quality System

Organizational support for implementing the quality system and achieving the quality objectives is derived from this LQM, SOPs and Work Instructions. Within these documents, management with executive responsibilities ensures that the quality policy is understood, implemented, and maintained at all levels of the organization. The development and implementation of appropriate accountabilities, duties, and authority by organizational positions are clearly delineated. Line organizations achieve and verify that specifications are achieved; QA organizations assist and provide oversight and verification of processes through planning, reviews, audits, and surveillances. Top management leadership, support and direction ensures that the policies and procedures are appropriately implemented.

4.2.1 Objectives of the Quality System

The goal of the quality system is to ensure that business operations are conducted with the highest standards of professionalism in the industry.

To achieve this goal, it is necessary to provide our clients with not only scientifically sound, well documented, and regulatory compliant data, but also to ensure that we provide the highest quality service available in the industry with uncompromising data integrity. A well-structured and well-communicated quality system is essential in meeting this goal. The laboratory's quality system is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

As stated in Section 1.3, this LQM, Work Instructions and the SOPs themselves are the basis and outline for our quality and data integrity system and contains requirements and general guidelines under which the laboratory conducts our operations. In addition, other documents may be used by the laboratory to clarify compliance with quality system or other client requirements. As you read this LQM, you will note SOP or Work Instruction numbers in parenthetic text. These numbers refer to the laboratory procedure(s) associated with the subject item. A table listing these quality system policies and procedures is appended to this document.

The QA Manager and QA Specialist are responsible for implementing and monitoring the Quality System. The QA Manager reports to the Laboratory Director on the performance of the quality system for review and continuous improvement. The QA Manager has sufficient authority, access to work areas, and organizational freedom (including sufficient independence from cost and schedule considerations) to:

- Initiate action to prevent the occurrence of any nonconformities related to product, process and quality system,
- Identify and record any problems affecting the product, process and quality system,
- Initiate, recommend, or provide solutions to problems through designated channels,
- Verify implementation of solutions, and
- Assure that further work is stopped or controlled until proper resolution of a non-conformance, deficiency, or unsatisfactory condition has occurred and the deficiency or unsatisfactory condition has been corrected.

The QA Manager reports where appropriate action can be affected. However, should a situation arise where acceptable resolution of identified problems cannot be agreed upon at the laboratory level, direct access to STL's Corporate Quality Director is available. This provides laboratory QA personnel non-laboratory management support, if needed, to ensure that QA policies and procedures are enforced.

The QA Manager or QA Specialist conducts annual LQM training for all laboratory and administrative personnel to ensure their familiarity with the quality documentation and the implementation of the policies and procedures in their work.

4.3 Document Control

The laboratory maintains procedures to control documents and analytical data. Since intensive data is generated and this is our primary product, document control is inherently segregated from data control, as described further in Sections 4.3.1 and 4.3.2.

4.3.1 Document Control Procedure

Security and control of documents are necessary to ensure that confidential information is not distributed and that all current copies of a given document are from the latest applicable revision (*Document Control*; UQA-006). Unambiguous identification of a controlled document is maintained by identification of the following items in the document header: Document Number, Revision Number, Effective Date, and Number of Pages. Document control may be achieved by either electronic or hardcopy distribution.

Controlled documents are authorized by the QA Department and are marked as either "Controlled' or "Uncontrolled" and records of their distribution are kept by the QA Department. Controlled status is defined as the continuous distribution of document updates. Uncontrolled status is defined as the single distribution of the current SOP. Document updates are not distributed to uncontrolled status holders. For tracking purposes, a control copy number is assigned to documents distributed with a controlled status. All copy numbers are written or typed in red to easily identify the SOP as a controlled copy.

4.3.1.1 Document Revision

Changes to documents occur when a procedural change warrants a revision of the document. When an approved revision of a controlled document is ready for distribution, obsolete copies of the document are replaced with the current version of the document. The previous revision of the controlled document is stamped "ARCHIVED COPY" and is filed by the QA Specialist in the QA library. Only the most current revision is maintained electronically.

SOPs are updated on a 12-18 month basis, which is tracked by an established review schedule (*Approved SOP Listing*; CHI-22-09-SOP List). These reviews are conducted by the creator of the SOP and/or Department Manager, QA Specialist and/or QA Manager, and the Health and Safety Coordinator, all of whom provide the approval signature for each SOP.

4.3.2 Data Control

All raw data, such as bound logbooks, instrument printouts, magnetic tapes, electronic data, as well as final reports, are retained for a minimum period of 5 years. Such data may be maintained longer, as defined by client and project requirements. The procedure for archiving records and client or project specific requirements is contained in the *Record Retention and Purging* SOP (UDM-002).

Raw data and reports are documented and stored in a manner in which they are easily retrievable. The procedure for maintaining raw data records is briefly described below:

- Instrument print-outs for conventional inorganic parameters are filed by LabNet Batch Number. Inorganic Metals are filed by Instrument and Filename. Generally, current year and previous year documents are kept on file in the laboratory sections.
- All raw data, for example, instrument print-outs and logbooks, are maintained in an on-site and secured storage area.
- The computer information is backed up on tape daily, and stored in a secured and temperature/humidity controlled environment to maintain the integrity of the electronic information in the event of system failure. Copies of all back-up tapes are maintained in secured off-site locations.
- All copies of client final reports are maintained electronically (e.g., Adobe Acrobat).

4.4 Request, Tender, and Contract Review

4.4.1 Contract Review

For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is STL's intent to provide both standard and customized environmental laboratory services to our clients. To ensure project success, technical staff performs a thorough review of technical and QC requirements contained in contracts. Contracts are reviewed for adequately defined requirements and STL's capability to meet those requirements.

All contracts entered into by the laboratory are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity, accuracy, and precision requirements. The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The review also includes the laboratory's capabilities in terms of turnaround time, capacity, and resources to provide the services requested, as well as the ability to provide the documentation, whether hardcopy or electronic. If the laboratory cannot provide all services but intends to subcontract such services, whether to another STL facility or to an outside firm, this will be documented and discussed with the client prior to contract approval.

Any contract requirement or amendment to a contract communicated to STL verbally is documented and confirmed with the client in writing. Any discrepancy between the client's requirements and STL's capability to meet those requirements is resolved in writing before acceptance of the contract. Contract amendments, initiated by the client and/or STL, are documented in writing for the benefit of both the client and STL.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the permanent project record as defined in Section 4.12.1.

4.4.2 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, STL assigns a Project Manager (PM) to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project (*Project Planning Process*; UPM-003). QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the direct client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure that the available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project Technical Profile (e.g., LabNet Project Notes) turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through *Project Kick-Off Meetings (UPM-002)* or to the supervisory staff during *Production Meetings (UPM-004)*. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, the LabNet Project Notes are associated with each sample batch (e.g., Job) as a reminder upon sample receipt and analytical processing.

Any changes that may occur within an active project is agreed upon between the client/regulatory agency and the Project Manager/laboratory. These changes (e.g., use of a non-standard method or modification of a method) must be documented prior to implementation. Documentation pertains to any document, e.g., letter, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory through the management Production Meetings which are conducted three times per week (T,W,Th). Such changes are updated to the LabNet Project Notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the Project Manager or the individual laboratory section manager. After the modification is implemented into the laboratory procedure, documentation of the modification is made in the case narrative of the data report(s).

STL strongly encourages our clients to visit the laboratory and hold formal or informal sessions with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

4.4.3 Data Quality Objectives

Data quality objectives (DQO) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application. Typically, DQOs are identified before project initiation and during the development of a QAPPs and SAPs. The analytical DQOs addressed in this section are precision, accuracy, representativeness, completeness, and comparability.

The components of analytical variability (uncertainty) can be estimated when QC samples of the right types and at the appropriate frequency are incorporated into the measurement process of the laboratory. STL incorporates numerous QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The control samples and their applications, described in Section 5.8.2, are selected based on regulatory, method- or client-specific requirements. Analytical QC samples for inorganic and organic analyses may include calibration blanks, instrument blanks, method blanks, LCS, calibration standards, MS, MSD, MD, surrogate spikes, and yield monitors.

The DQOs discussed below ensure that data are gathered and presented in accordance with procedures appropriate for its intended use, that the data is of known and documented quality, and are able to withstand scientific and legal scrutiny.

4.4.3.1 Precision

Precision is an estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. Precision is expressed either as Relative Standard Deviation (RSD) for greater than two measurements or as Relative Percent Difference (RPD) for two measurements. Precision is determined, in part, by analyzing data from LCSs, MS, MSD, and MD. A description of these control samples is provided in Section 5.8.2.

Precision also refers to the measurement of the variability associated with the entire process, from sampling to analysis. Total precision of the process can be determined by analysis of duplicate or replicate field samples and measures variability introduced by both the laboratory and field operations.

4.4.3.2 Accuracy

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. It reflects the total error associated with a measurement.

Both random and systematic errors can affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100). Accuracy is determined, in part, by analyzing data from LCSs, MS and MSD.

Accuracy and Precision objectives employed by the laboratory are as defined in the CERCLA's Inorganic and Organic Statements of Work (SOW); statistically-derived control limits; or default limits as listed in each respective method SOP.

4.4.3.3 Representativeness

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition. Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result is representative of the constituent concentration in the sample matrix. STL makes every effort to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before sub-sampling.

4.4.3.4 Completeness

Completeness is defined as the percentage of measurements that are judged valid or useable. Factors negatively affecting completeness include the following: sample leakage or breakage in transit or during handling, loss of sample during laboratory analysis through accident or improper handling, improper documentation such that traceability is compromised, or sample result is rejected due to failure to conform to QC specifications. A completeness objective of greater than 90% of the data specified by the statement of work is the goal established for most projects.

4.4.3.5 Comparability

Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (e.g., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

A measure of inter-laboratory comparability is obtained through the laboratory's participation in proficiency testing (PT) programs established with Water Supply (WS), Water Pollution (WP), Solid Waste (SW), and Underground Storage Tank (UST) programs. In addition, the laboratory employs the use of NIST or EPA traceable standards, when available, to provide an additional measure of assurance of the comparability of data.

Project representativeness and comparability are dependent upon the sampling plan on a project specific basis, and are therefore not covered in this LQM. Assessment of site and collection representativeness and comparability is performed by the field engineer.

4.4.3.6 Additional DQOs

Method Detection Limits

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants". MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually. *(UQA-017)*

For the performance of non-routine methods, e.g., client/contract requirement, MDLs or Method Validation Studies will be completed on an as needed basis. The turnaround time for such studies will be as determined by the client and Project Manager. Such studies will be reviewed and approved by the client and/or regulatory agency prior to project implementation.

Instrument Detection Limits

There are a number of ways to determine Instrument Detection Limit (IDL) sensitivity (e.g., signal-to-noise ratio; precision of the low-level standard; lowest calibration curve point or the IDL study defined within CLP). The method and means in which IDLs are determined are documented and maintained in the QA department for each individual instrument.

IDLs are generated for each element by the metals laboratory quarterly via each instrument as specified in CLP. These limits are used to gauge instrument sensitivity and when routinely evaluated, instrument performance without the introduction of method variance can be determined. *(UQA-010)*

Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory reporting limits are further related and verified by the lowest point on a calibration curve. Because of the high level of quantitative error associated with determinations at the level of the MDL, the laboratory maintains reporting limits higher than the MDL. Wherever possible, reporting is limited to values approximately 2-5x the respective MDL to ensure confidence in the value reported. Client specific requests for reporting to the IDL or MDL are special circumstances not to be confused with the previous statement. Data evaluated down to the MDL/IDL is qualified as estimated with a 'J' for organic analyses and a 'B' for inorganic analyses on the data report.

MDL studies are performed annually, and reporting limits are assessed. If the MDL does not meet the routine laboratory reporting limit or the method specified limit, it is repeated or the laboratory reporting limit is reassessed. If the laboratory continually demonstrates that the method reporting limits are not achieved, equipment, technique, and the method are reviewed to assure optimal performance or appropriate action is taken.

4.5 Subcontracting

Subcontracting is arranged with the documented consent of the client, in a timely response which shall not be unreasonably refused. All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Proof of holding required certifications from the subcontract facility are maintained in the project records. Where applicable, the specific QC guidelines, QAPPs, and/or SAPs are transmitted to the subcontract laboratory. Samples are subcontracted under formal Chain of Custody (COC).

Subcontract laboratories may receive an on-site audit by a representative of STL's QA staff if it is deemed appropriate by the QA Manager. The audit involves a measure of compliance with the required test method, QC requirements, as well as any special client requirements (e.g., Technical Profile and LabNet Project Notes). STL may also perform a paper audit of the subcontractor, which would entail reviewing the LQM, the last two PT studies, and a copy of any recent regulatory audits with the laboratory's responses.

Intra-company subcontracting may also occur between STL facilities. Intra-company subcontracting within STL is arranged with the documented consent of the client (e.g., QAPP). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. STL has implemented a standard form for Intra-laboratory subcontracting, refer to the following document for specific details: *Work Sharing Process – Policy No.: S-C-001.*

Project reports from both STL and external subcontractors are not altered and are included in their original form in the final project report provided by STL. This clearly identifies the data as being produced by a subcontractor facility. All data, as required in Section 5.9.4, is included.

4.6 Purchasing Services and Supplies

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specific requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff.

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents meet with the requirements of the specific method and testing procedures for which they are being purchased. The measurements for evaluation and selection of suppliers; the acceptance of supplies and services; and certificates of conformance are described in the procurement SOP (*Procurement Quality Assurance Process*; UQA-020).

4.6.1 Solvent and Acid Lot Verification

Pre-purchase approval is performed for solvents and acids purchased in large quantities unless a certificate of conformance has been furnished. These may include acetone, ethyl ether, hexane, methylene chloride, nitric acid, hydrochloric acid, sulfuric acid, and hydrogen peroxide. Each lot of incoming supplies requiring pre-approval is checked against the previously approved lot number. If the lot number is not approved, the lot is refused. If the lot number is an approved lot number, it is accepted and documented. Solvents and acids are pre-tested in accordance with STLs Corporate *Testing Solvents and Acids* procedure (S-T-001) for all of the STL laboratories.

4.7 Service to the Client

4.7.1 Sample Acceptance Policy

Samples are considered "compromised" if the following conditions are observed upon sample receipt:

- Cooler and/or samples are received outside of temperature specification.
- Samples are received broken or leaking.
- Samples are received beyond holding time.
- Samples are received without appropriate preservation.
- Samples are received in inappropriate containers.
- COC does not match samples received.
- COC is not properly completed or not received.
- Breakage of any Custody Seal.
- Apparent tampering with cooler and/or samples.
- Headspace in volatiles samples.
- Seepage of extraneous water or materials into samples.
- Inadequate sample volume.
- Illegible, impermanent, or non-unique sample labeling.

When "compromised" samples are received, it is documented on the hardcopy COC, the LabNet Sample Receipt Checklist and on a Sample Discrepancy Report (SDR); and the client is contacted for instructions. If the client decides to proceed with the analysis, the project report will clearly indicate any of the above conditions and the resolution.

4.7.2 Client Confidentiality and Proprietary Rights

Data and sample materials provided by the client or at the client's request, and the results obtained by STL, shall be held in confidence (unless such information is generally available to the public or is in the public domain or client has failed to pay STL for all services rendered or is otherwise in breach of the terms and conditions set forth in the STL and client contract) subject to any disclosure required by law or legal process. Technical, business and proprietary information provided by a client and data/information generated by the laboratory are restricted for the use within the laboratory for purposes of accomplishing the project. Client information is not to be used on other projects or revealed except in conjunction with project work to anyone outside the laboratory without permission of the client.

STL's reports, and the data and information provided therein, are for the exclusive use and benefit of client, and are not released to a third party without written consent from the client (*Client Confidentiality*; UQA-004).

4.8 Complaints

STL believes that effective client complaint handling processes have important business and strategic value. Listening to and documenting client's concerns captures 'client knowledge' that helps to continually improve processes and outpace the competition. Implementing a client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly. The investigation of the cause, resolution and authorization of corrective action is documented [*Sample Discrepancy Report (SDR), Resubmitted Data Request (RDR), Corrective Action Report (CAR)*; UQA-029].

Client complaints are documented by the employee receiving the complaint. The documentation can take the form of a Resubmitted Data Request (RDR) or in a format specifically designed for that purpose (e.g., phone conversation record or e-mail). The Laboratory Director, Project Manager and/or QA Manager are informed of client complaints and assist in resolving the complaint.

The RDR is used after the client has received the analytical report and their specifications, expectations, or client satisfaction was not achieved. RDRs are prepared when clients request re-evaluation of submitted data, when additional information is requested or for general complaints.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA department is required to conduct a special audit to assist in resolving the issue. A written confirmation, or letter to the client outlining the issue and response taken, is strongly recommended as part of the overall action taken.

The number and nature of client complaints is reported by the QA Manager to the QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the *Quality Systems Management Review* (UQA-002).

4.9 Control of Non-conformances

Non-conformances include any out of control occurrence. Non-conformances may relate to client specific requirements, procedural requirements, or equipment issues. All non-conformances in the laboratory are documented at the time of their occurrence on Corrective Action Reports (CARs) specifically formatted for each department or on a SDR.

All non-conformances that affect a sample and/or sample data become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Section Manager, Project Manager or QA Manager for direction may be required. All records of reanalysis are kept with the project files.

Where non-conformances specifically affect a client's sample and/or data, the client is informed and action must be taken. Action can take the form of reporting and flagging the data, and including a description of the non-conformance in the project narrative.

4.10 Corrective Action

To consistently achieve technical and regulatory requirements, the laboratory data must be supported by an effective corrective action system. The system must be capable of isolating and rectifying both random and systematic errors. Identification of systematic errors, or errors that are likely to occur repetitively due to a defect or weakness in a system, is particularly valuable in maintaining an environment of continuous improvement in laboratory operations.

Mechanisms used to ensure problem definition include SOPs; internal and external audits and surveillances; and regular laboratory management meetings. When evaluation of performance against established criteria for good laboratory practices shows a condition that could adversely affect the quality of services provided, corrective action is initiated.

Any employee in STL can initiate a corrective action. The initial source of corrective action can also be external to STL (i.e., corrective action due to client complaint, regulatory audit, or PT(s)). When a problem that requires corrective action is identified, the following items are identified by the initiator on the corrective action report: the nature of the problem, the name of the initiator, and the date. If the problem affects a specific client project, the PM is informed immediately.

All corrective actions, whether immediate or long-term, will comprise the following steps to ensure a closed-loop corrective action process:

- Define the problem.
- Assign responsibility for investigating the problem.
- Determine a corrective action to eliminate the problem.
- Assign, and obtain commitment to, responsibility for implementing the corrective action.
- Implement the correction.
- Assess the effectiveness of the corrective action and verify that the corrective action has eliminated the problem.

4.10.1 Immediate Corrective Action

Immediate corrective actions to correct or repair non-conforming equipment and systems are generally initiated in response to adverse conditions identified through QC procedures. The analyst has relatively quick feedback that a problem exists, e.g., calibration does not meet or QC check samples exceed allowable criteria, and can take immediate action to repair the system.

The initial responsibility to monitor the quality of a function or analytical system lies with the individual performing the task or procedure. DQOs are evaluated against laboratory-established or against method or client specified QA/QC requirements. If the assessment reveals that any of the QC acceptance criteria are not met, the analyst must immediately assess the analytical system to correct the problem. When the appropriate corrective action measures have been defined and the analytical system is determined to be "in-control" or the measures required to put the system "in-control" have been identified and scheduled, the problem and resolution or planned action is documented in the appropriate logbook or CAR. Data generated by an analytical system that is determined to be out-of-control must never be released without approval of the Section Manager, QA Manager, Laboratory Director, Project Manager and client notification.

When an acceptable resolution cannot be met or data quality is negatively affected, the analyst will notify their Section Manager and initiate an SDR. If an SDR is required, it is routed for proper authorizations and direction. Proper authorization and direction is given by the Project Manager and/or QA Manager. Based upon the circumstances and judgment of the Project Manager, the client may be notified of the situation.

Data generated concurrently with an out-of-control system will be evaluated for usability in light of the nature of the deficiency. If the deficiency does not impair the usability of the results, data will be reported and the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by a written SDR and appropriate corrective action (e.g., reanalysis) is taken and documented.

A CAR documents analytical problems at the bench level. This form allows for the documentation of the out-of-control situation, actions undertaken to correct the problem and a return-to-control status. All CARs are signed/dated by the respective laboratory Section Manager.

The QA Manager has the authority to stop the analysis, e.g., failure to meet method or project requirements, and to hold all analyses of samples affected by an out-of-control situation. The method cannot be restarted without appropriate documentation leading to the QA Manager's approval and sign-off.

4.10.2 Long-term Corrective Action

Long-term corrective action is generally initiated due to QA issues, which are most often identified during internal and external audits (Sections 4.13 & 4.14). Typically, a deeper investigation into the root cause of the nonconformance is warranted, and the problem may take much longer to identify and resolve. Staff training, method revision, replacement of equipment, and LabNet reprogramming are examples of long-term corrective action.

4.10.3 Responsibility and Closure

The Section Manager is responsible for correcting out-of-control situations, placing highest priority on this endeavor. Associated corrective actions, once verified for effectiveness, are incorporated into standard practices. Ineffective actions will be re-evaluated until acceptable resolution is achieved. Section Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved.

The QA Department also may implement a special audit (Section 4.13). The purpose of inclusion of the corrective action process in both routine and special audits is to monitor the implementation of the corrective action and to determine whether the action taken has been effective in overcoming the issue identified.
Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation. This provides laboratory QA personnel non-laboratory management support, if needed, to ensure that QA policies and procedures are enforced.

4.11 Preventative Action

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity which can be initiated by clients, employees, business providers, and affiliates. The QA section has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Preventive action opportunities may be identified from information obtained through activities related to but not limited to the corrective action process, performance evaluation program, internal audits, management review, and/or market trends, industry trends and competitive comparisons.

Established standard practices for preventive action are included in the *Preventive Action Measures* SOP (UQA-019); the *SDR / RDR / CAR* SOP (UQA-029) and the *Quality System Management Review* SOP (UQA-002). These procedures describe the information sources used to detect, analyze, and eliminate potential causes of nonconformities and to ensure effective implementation of solutions.

4.12 Records

4.12.1 Record Types

Record types are described in Table 4.

4.12.2 Record Retention

Data reports are filed electronically as .pdf files by sample job number. Hardcopy COC files are maintained and are filed in Job Number order.
Laboratory data, project management files, QA records (e.g., PT scores/corrective actions; MDLs/IDLs, statistical analysis, QAPPs, etc.), Human Resources information, etc.., are compiled by date order. The same procedure is followed both in current and archived hardcopy storage.

Upon archiving, a *Records Management Form* (CHI-22-05-032) is prepared for each storage box of records. This form documents the department, department manager, contents (description and dates), term of retention (e.g., no. of years) and an assigned identification number. The original of this form is maintained with the data management department with a carbon copy filed within the storage box. Upon purging of records, the individual department managers sign the original form as confirmation for the destruction of the associated data. This signature indicates that the laboratory has maintained the information for the required amount of time and is no longer required to store it.
Table 5 outlines the laboratory's standard record retention time. For raw data and project records, record retention is calculated from the date the project report is issued. For other records, such as Controlled Documents, QC, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 6 have lengthier retention requirements and are subject to the requirements in Section 4.12.3.

Table 4. STL Record Types

Raw Data	Controlled Documents	QC Records	Project Records	Administrative Records
See Section 3. Terms and Definitions	LQMs/ QAPPs	Audits/ Responses	COC Documentation	Accounting
	QMP (Corporate)	Certifications	Contracts and Amendments	Corporate Safety Manual, Permits, Disposal Records
	SOPs	SDRs/RDRs	Correspondence	Employee Handbook
		Logbooks*	QAPP	Personnel files, Employee Signature & Initials, Training Records
		Method & Software Validation, Verification	SAP	
		Standards Certificates	Telephone Logbooks	Technical and Administrative Policies
	Work Instructions	MDL/IDL/IDC Studies	E-mails	
		PTs	Electronic Data Report	
		Statistical Evaluations		

*Examples of Logbook types: Maintenance, Instrument, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, and Balance Calibration.

Table 5. STL Record Retention

Record Type		Archival Requirement *
Raw Data	All* (Electronic Data Reports (.pdf & EDD)	5 Years from completion
Controlled Documents	All*	5 Years from document retirement date
QC	All*	5 Years from archival
Project	All*	5 Years from project completion
Administrative	Personnel/Training	Indefinitely
	Accounting	10 years

* Exceptions listed in Table 6.

4.12.3 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the laboratory's standard record retention time. These are detailed in Table 6 with their retention requirements and client-specific requirements are listed in the *Record Retention and Purging* SOP (UDM-002). In these cases, the longer retention requirement is implemented and noted in the archive. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 6. Special Record Retention Requirements

Program	Retention Requirement
Colorado – Drinking Water	10 years
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Massachusetts – Drinking Water	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Minnesota – Drinking Water	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
OSHA - 40 CFR Part 1910	30 years
Pennsylvania – Drinking Water	10 years
TSCA – 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

4.12.4 Archives and Record Transfer

Archives are indexed such that records are accessible on either a project or temporal basis. Archives are protected against fire, theft, loss, deterioration, and vermin. Electronic records are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to archives is controlled and documented.

STL ensures that all records are maintained as required by the regulatory guidelines and per this LQM upon facility location change or ownership transfer. Upon facility location change, all archives are retained by STL in accordance with this LQM. Upon ownership transfer, all final test reports generated by the laboratory will be submitted to the clients if not previously provided. Any further record retention requirements will be addressed in the ownership transfer agreement and the responsibility for maintaining archives will be clearly established.

In the event that the laboratory is closed, all final test reports generated by the laboratory will be submitted to the clients if not previously provided. All records will then be transferred to STL's corporate record storage location. All boxes and contents will be appropriately labeled with the dates of destruction (Refer to Tables 5 and 6) and managed in accordance their policies.

4.13 Internal Audits

Quality assurance audits and surveillances are conducted to assess the performance of laboratory systems in meeting technical, regulatory and client requirements; and to evaluate the operational details of the QA program (*Internal Audits*; UQA-013). They provide a means for management to be apprised of, and to respond to, a potential problem before it actually impacts the laboratory operations. They also are a mechanism for ensuring closure of corrective actions resulting from external audits.

4.13.1 Audit Types and Frequency

A number of types of audits are performed at STL. These audit types and frequency are categorized in Table 7.

Table 7. Audit Types and Frequency

Audit Type	Performed by	Frequency
Systems	QA Department or Designee	Annual
Data Authenticity	QA Department or Designee	**Data Report Review:** As necessary to ensure an effective secondary review process and to meet special program independent review objectives
		Analyst Data Audits: 100% of all analysts annually
Electronic		**Electronic Data Audits:** 100% of all organic instruments
Special	QA Department or Designee	As Needed

4.13.2 Systems Audits

Systems audits are technical in nature and are conducted on an ongoing basis by the QA Manager or the QA Specialist. Systems audits cover all departments of the facility, both operational and support. The review consists of laboratory systems, procedures, documentation and issues noted in external audits.

The audit report is issued by the QA Manager or QA Specialist within 21 calendar days of the audit. The audit report is addressed to the department Section Manager and copied to the QA department and the Laboratory Director.

Written audit responses are required within 30 calendar days of the audit report issue. A maximum of one calendar month is given to address any recommended corrective actions. The audit response is directed to all individuals copied on the audit report. Where a corrective action may require longer than a calendar month to complete, the target date for the corrective action implementation is stated and evidence of the corrective action is submitted to the QA Department in the agreed upon time frame.

4.13.3 Data Audits

Data audits are focused to assess the level of customer service, SOP compliance, regulatory compliance, accuracy and completeness of test results and reports, documentation, and adherence to established QC criteria, laboratory SOPs, technical policy, and project specific QC criteria.

The QA Department provides feedback and/or corrections and revisions to project reports where necessary. Records of the data audits are kept, and the frequency of data audits is included in the monthly QA report. In performing data audits, it is essential that data be assessed in terms of differentiating between systematic and isolated errors. Upon noting anomalous data or occurrences in the data audits, the QA Department is responsible for seeking clarification from the appropriate personnel, ascertaining whether the error is systematic or an isolated error, and overseeing correction and/or revision of the project report if necessary. Errors found in client project reports are revised and the revision sent to the client (Section 4.8). The QA Department is also responsible for assisting in the corrective action process where a data audit leads to identification of the need for permanent corrective action.

The frequency of data auditing may also be dependent upon specific clients and regulatory programs. All active laboratory logbooks and QC files are subject to periodic audits/ surveillances by the QA personnel.

4.13.3.1 Data Authenticity Audits

Data authenticity audits shall be performed on 100% of all analysts by the QA department or a designee independent from laboratory operations. Performing data authenticity checks will typically include verifying raw data, evaluating calculation tools and independently reproducing the final results and comparing it to the hardcopy on randomly selected batches of data. The QA Manager will report the percentage of analysts reviewed (for the year) in the monthly QA report and should average about 8% per month.

4.13.3.2 Electronic Data Audits

Electronic data audits are performed on 100% of all organic instruments by the QA department or a designee independent from the operations. This may include Mint Miner® scanning of randomly selected batches of electronic data followed by a chromatography system review. The QA manager will report the percentage of instruments reviewed (for the year) in the monthly QA report and should average about 8% of instruments per month. Electronic data audits include spot-checking of manual integrations by QA personnel in order to determine that the manual integration is appropriate and documented according to Section 5.3.6.1.

4.13.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, proficiency testing results, data audits, systems audits, validation comments, or regulatory audits. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

4.14 External Audits

STL is routinely audited by clients and external regulatory authorities – both government and non-government. Whether the audit is scheduled or unannounced, full cooperation with the audit team is provided by the laboratory and administrative staff. STL recommends that the audits be scheduled with the QA Department so that all necessary personnel are available on the day of the audit.

4.15 Management Reviews

4.15.1 QA Reports to Management

A monthly QA report is prepared by QA Manager and forwarded to the Laboratory Director, Project Managers, Section (Technical) Managers and the Corporate Quality Director. The reports include statistical results that are used to assess the effectiveness of the quality system. The format of the monthly report is shown in Figure 3.

4.15.2 Quality Systems Management Review

A quality systems management review is performed at least annually by the QA Manager. This review ensures that the laboratory's quality system is adequate to satisfy the laboratory's policies and practices, government requirements, certification, accreditation, approval requirements, and client expectations. Quality systems management reviews are accomplished through the evaluation and revision of this LQM, monthly quality assurance reporting and goal setting.

Management reviews of specific quality system elements may be performed through continuous improvement activities, monthly QA reports, process changes, SOP revisions, and/or audit reports/responses. Documentation of these reviews are not required unless it is inherent in the review mechanism (e.g., approval signatures on SOP revisions).

4.15.3 Monthly QA Report and Metrics

By the 3rd day of the month, the QA manager prepares a monthly QA report. The report is sent to the Laboratory Director and Corporate Quality Director. The report contains a narrative summary and metrics spreadsheet. At a minimum, the report content contains the items listed below (Figure 3). During the course of the year, the Laboratory Director, General Manager or Corporate Quality Director may request that additional information be added to the report.

Figure 3. Monthly QA Report Format

1	Audits
	External System Audits Internal System Audits Internal Training Record Audits Internal Data Audits
2	Revised Reports / Client Complaints / Client Compliments
	Revised Reports (RDR) Client Complaints Client Compliments
3	Certification Changes
	Certification Status Losses / Revocations
4	Proficiency Testing
	Study participation PT scores PT failures History of failures
5	SOP Status
	SOPs totals summarized by manager On-Time percentages calculated for SOPs < 1 year
6	Project/QAPP Review Status
7	Holding Time Violations
8	Monthly QA Report Metrics
	Summarize metrics in template provided by the Corporate Quality Director

5.0 Technical Requirements

5.1 Personnel

5.1.1 General

STL management believes that its highly qualified and professional staff is the single most important aspect in assuring the highest level of data quality and service in the industry. The staff consists of professionals and support personnel that include the following positions:

- Laboratory Director
- QA Manager
- Health & Safety Coordinator / Waste Management
- Project Manager
- Information Technology Manager
- Department Section Manager (Technical Manager)
- Analyst
- Sample Custodian
- Technician
- Quality Assurance Specialist
- Data Review Specialist

In order to ensure that employees have sufficient education and experience to perform a particular task, job descriptions are developed for all personnel (Section 4.1.2).

5.1.2 Training

STL is committed to furthering the professional and technical development of employees at all levels. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for STL employees are outlined in Table 8.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. The QA department, in conjunction with the Human Resources coordinator and Section Supervisor are responsible for maintaining the documentation of these activities.

Each laboratory section maintains documentation associated with analytical training (e.g., training records, document control). The QA department maintains documentation of initial and continued method proficiency for laboratory instrumentation and for each analyst. This documentation is represented in the following forms: MDLs, IDMPs, IDOCs, CDOCs, PT Sample results, Instrument QC and Batch QC Control Charts. This information is available to managers and staff for planning and evaluation.

The Human Resource coordinator maintains documentation and attestation forms on employment status & records; benefit programs; time keeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

The following evidence items are on file for each technical employee:

- Initial Demonstration of Capability (IDOC) for each method.
- Attestation that the employee has read and understood the latest version of the laboratory's quality documentation.
- The employee has read and understood the latest, approved version of all test methods and/or SOPs for which the employee is responsible.
- Annual evidence of Continued Demonstration of Capability (CDOC) that may include, but is not limited to, successful analysis of a blind sample on the specific test method or a similar test method; an annual DOC of four successive and acceptable LCSs.
- An Ethics Agreement signed by each staff member (renewed each year).
- A Confidentiality Agreement signed by each staff member (renewed each year).

Table 8. STL Employee Minimum Training Requirements

Specialty	Experience
General Chemistry and Instrumentation	Six months
Gas Chromatography	One year
Atomic Absorption	One year
Mass Spectrometry	One year
Spectra Interpretation	Two years

Required Training	Time Frame [1]	Employee Type
Environmental Health & Safety	Month 1	All
Ethics – Corporate Overview	Week 1	All
Ethics Data Integrity	Month 1 Month 1	All Technical and PMs
Ethics Refresher	Annually	All
Quality Assurance	Quarter 1	All
Initial Demonstration of Capability (IDOC)	Prior to unsupervised method Performance	Technical

[1] From the date of initial employment unless otherwise indicated.

The quality assurance training includes an overview of regulatory programs and program goals, a review of the ethics statement, and group discussions about data integrity and data misrepresentation.

When an analyst does not meet these requirements, they can perform a task under the supervision of a qualified analyst, peer reviewer or section manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

IDOCs (Initial Demonstration of Method Capability) are performed by the analysis of four replicate QC samples. Results of successive LCS analyses can be used to fulfill the IDOC requirement, however, LCSs performed over several batches is desirable. The accuracy and precision, measured as average recovery and standard deviation (using n-1 as the population), of the 4 replicate results are calculated and compared to those in the test method (where available). If the test method does not include accuracy and precision requirements, the results are compared to target criteria set by the laboratory. The laboratory sets the target criteria such that they reflect the DQOs of the specific test method or project. An IDOC Certification Statement is recorded and maintained in the employee's training file. Tabulated results summary and raw data are completed and signed by the analyst and section manager with the proper entries made onto the analysts training record. The data is submitted to the QA department for approval and entry into the master IDOC spreadsheet and for filing. Figure 4 shows an example of an *IDOC Certification Statement.* (CHI-22-09-271)

On an annual basis, the analyst's method capabilities must be evaluated. The requirement that a CDOC (Continued Demonstration of Capability) be completed for each method currently being analyzed must be presented for approval to the QA department. (e.g. *Yearly Method Capability Review Work Instruction-Wet Chemistry*: CHI-22-09-279)

Further details of the laboratory's training program are described in the Laboratory Training SOP (UQA-014).

Figure 4. Demonstration of Capability Certification Statement

Demonstration of Capability
Certification Statement

Date:
STL Chicago
2417 Bond Street
University Park, IL 60466

Analyst Name:________________
SOP No.:________________
Method No.:________________
Description:________________
Matrix:________________
Effective Date:________________

We the undersigned certify that:

1. The analyst identified above, using the cited test method(s), which is in use at this laboratory for the analysis of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.
2. The test method(s) was performed by the analyst identified on this certification.
3. A copy of the reference method and laboratory-specific SOP(s) are available for all personnel on-site.
4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory.
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the laboratory, and that the associated information is well organized and available for review by authorized assessors.

Technical Manager	Signature	Date	
Quality Assurance Manager	Signature	Date	

5.1.3 Ethics Policy

Establishing and maintaining a high ethical standard is an important element of a Quality System. In order to ensure that all personnel understand the importance the company places on maintaining high ethical standards at all times; STL has established an Ethics Policy (P-L-006) and an Ethics Agreement (Figure 5). Each employee signs the Ethics Agreement, signifying agreed compliance with its stated purpose on an annual basis.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize the Company's ability to do work on Government contracts, and for that reason, the Company has a Zero Tolerance approach to such violations.

Ethics is also a major component of STL's quality and data integrity systems. Each employee is trained in ethics within thirty days of hire and quality training within three months of hire. Annual ethics refresher training will be provided. Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. A data integrity hotline is maintained by STL and administered by the Corporate Quality Director.

Figure 5. STL Ethics Agreement

I understand that STL is committed to ensuring the highest standard of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.

With regard to the duties I perform and the data I report in connection with my employment at the Company, I agree that:

- I will not intentionally report data values that are not the actual values obtained;
- I will not intentionally report the dates, times, sample or QC identification, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations;
- I will not intentionally misrepresent another individual's work;
- I will not intentionally report data values that do not meet established quality control criteria as set forth in the Method and/or Standard Operating Procedures, or as defined by Company Policy;
- I agree to inform my Supervisor of any accidental reporting of non-authentic data by me in a timely manner; and I agree to inform my Supervisor of any accidental or intentional reporting of non-authentic data by other employees; and
- If a supervisor or a member of STL management requests me to engage in or perform an activity that I feel is compromising data validity or quality, I will not comply with the request and report this action immediately to a member of senior management, up to and including the President of STL.

As a STL employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.

The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination. In addition, I understand that any violation of this policy which relates to work under a government contact or subcontract could also subject me to the potential for prosecution under federal law.

EMPLOYEE SIGNATURE:______________________________ Date: ________________
Supervisor/Trainer:______________________________ Date:________________

5.2 Facilities

The laboratory is a secure facility with controlled and documented access. Access is controlled by various measures including locked doors, electronic access cards, security codes, and a staffed reception area. All visitors sign in and are escorted by STL personnel while at the facility. The laboratory is locked at all times, unless a receptionist is present to monitor building access (e.g., between the hours of 8:00 a.m. and 5:00 p.m. Monday through Friday).

The facility is designed for efficient, automated high-quality operations. The laboratory is equipped with Heating, Ventilation, and Air Conditioning (HVAC) systems appropriate to the needs of environmental testing laboratories. Environmental conditions in the facility, such as hood flow, are routinely monitored and documented.

The facility is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. STL also provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc..

5.3 Test Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices.

5.3.1 Method Selection

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager in a Technical Profile and within LabNets Project Notes feature. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists, etc..), the method of choice is selected based on client needs and available technology.

Most of the test methods performed at STL originate from test methods published by a regulatory agency such as the US EPA and other state and federal regulatory agencies. These include, but are not limited to, the following published compendiums of test methods. A listing of methods in which the laboratory is capable of performing is listed in laboratory's *Methods Capabilities* Work Instruction (CHI-22-09-255).

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water.

Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-003, February 1999.

Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.

Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.

Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.

NIOSH Manual of Analytical Methods, 4th ed., August 1994.

Statement of Work for Inorganics Analysis, ILM04.0, USEPA Contract Laboratory Program Multi-media, Multi-concentration.

Statement of Work for Organics Analysis, OLM04.2 and OLC02.1, USEPA Contract Laboratory Program, Multi-media, Multi-concentration.

Standard Methods for the Examination of Water and Wastewater, 18th/19th/20th edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc.., and establishes an implementation schedule. As such, the laboratory strives to perform only the latest versions of each approved method.

5.3.2 SOPs

STL maintains an *Approved SOP Listing* (CHI-22-09-SOP) for both Method and Process SOPs. Method SOPs are maintained to describe a specific test method. Process SOPs are maintained to describe function and processes not related to a analytical testing (e.g., administrative procedures).

Method SOPs contain the following information:

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 6).

1. Identification of Test Method
2. Applicable Matrix
3. Scope and Application, including test analytes
4. Summary of the Test Method
5. Reporting Limits
6. Definitions
7. Interferences
8. Safety
9. Equipment and Supplies
10. Reagents and Standards
11. Sample Collection, Preservation and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Calculations
16. Method Performance
17. Pollution Prevention
18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Corrective Actions for Out-of-Control Data
20. Contingencies for Handling Out-of-Control or Unacceptable Data
21. Waste Management
22. References
23. Tables, Diagrams, Flowcharts and Validation Data

Process SOPs contain the following information:

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 6).

1. Scope
2. Summary
3. Definitions
4. Responsibilities
5. Procedure
6. References
7. Tables, Diagrams, and Flowcharts

The QA Department is responsible for maintenance of SOPs, archival of SOP historical revisions, maintenance of an SOP index, and records of controlled distribution. SOPs, at a minimum, undergo annual review (12-18 months). Where an SOP is based on a published method, the laboratory maintains a copy of the reference method.

Figure 6. Proprietary Information Statement

This documentation has been prepared by Severn Trent Laboratories (STL) solely for STL's own use and the use of STL's customers in evaluating its qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to STL upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF STL IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY STL IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

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SOP Change Form

The SOP Change Form is used for implementation, documentation, and authorization of changes to SOPs (*SOP Change Protocol*; UQA-032). Immediate changes in SOPs may be necessary to accommodate improvements; to implement acceptable changes in practices; or to correct potential errors in the existing version. The reason for the change will be identified and a detailed description of the procedure change will be presented. Since this form will become part of the referenced SOP, until such time that the SOP is updated, it must be legible and comprehensible. The Change Form must provide an exact description and identify the affected sections.

Once this form is completed and changes are authorized, it becomes an official part of the SOP for which it revises, and is subject to all document control and records management policies.

5.3.3 Method Validation

Laboratory developed methods are validated and documented according to the procedure described in Section 5.3.5.

5.3.4 Method Verification

Method verification is required when a validated standard test method or a method modification is implemented. The level of activity required for method verification is dependent on the type of method being implemented, or on the level of method modification and its affect on a method's robustness. Method modification often takes advantage of a method's robustness, or the ability to make minor changes in a method without affecting the method's outcome.

It is the responsibility of the section manager to present to the QA manager all applicable method validation studies for review and approval. The documented approval by the section manager and QA manager must be applied to all applicable validation records before the method is released for use. Method verification may require some, but not all, of the activities described in Section 5.3.5.

5.3.5 Method Validation and Verification Activities

Before analyzing samples by a particular method, method validation and/or method verification must occur. A complete validation of the method is required for laboratory developed methods. While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

Determination of Method Selectivity
Method selectivity is demonstrated for the analyte(s) in the specific matrix or matrices. In some cases, to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

Determination of Method Sensitivity
Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed. The laboratory determines MDLs are described in Section 4.4.3.6 and within UQA-017 and the corporate procedure S-Q-003.

Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)
An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum level at which both the presence of an analyte and its concentration can be reliably determined. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

Determination of Interferences
A determination that the method is free from interferences in a blank matrix is performed.

Determination of Range
Where appropriate, a determination of the applicable range of the method may be performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of quantitation

and the lower and upper values of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

Demonstration of Capability
DOCs are performed prior to method performance.

Determination of Accuracy and Precision
Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

Documentation of Method
The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Appendix describing the specific differences in the new method is acceptable in place of a separate SOP.

Continued Demonstration of Method Performance
Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS and Method Blanks.

5.3.6 Data Reduction and Review

Analytical data are entered/downloaded directly into LabNet or recorded on pre-formatted bench sheets that are paginated and bound into laboratory logbooks. These logbooks are issued and controlled by the laboratory's QA Section. A unique document control code is assigned to each book to assure that chronological record keeping is maintained. Analytical data may be electronically stored as a secure .pdf file to which the analyst applies an electronic signature.

Analytical data is referenced to a unique sample identification number for internal tracking and reporting. Both LabNet entries and logbook pages contain the following information, as applicable: analytical method, analyst, date, sequential page number, associated sample numbers, standard concentrations, instrument settings, and raw data. Entries are in chronological order and maintained so as to enable reconstruction of the analytical sequence.

The analyst is responsible for entering / recording all appropriate information, and for signing and dating all logbook entries daily. All entries and logbook pages are reviewed for completeness by a supervisor, peer reviewer or the analyst themselves. Data review checklists document the analytical review of the LabNet entries, logbook and associated QC indicators. Copies of instrument outputs (chromatograms, mass spectra, etc..) are maintained on file or electronically with the analyst's signature/initials and date.

5.3.6.1 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the section manager or alternate analyst prior to updating the data in LabNet. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the STL Corporate SOP entitled *Acceptable Manual Integration Practices* (S-Q-004).

Copies of all raw data and the calculations used to generate the final results, such as bound logbooks, are retained on file for a minimum of 5 years or as otherwise requested by the client/project.

Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

5.3.6.2 Data Review

All data, regardless of regulatory program or level of reporting, are subject to a thorough review process. The individual analyst continually reviews the quality of the data through calibration checks, quality control sample results and performance evaluation samples. Data review is initiated by the analyst during, immediately following, and after the completed analysis.

All levels of the review are documented on Data Review Checklists that are specific to each laboratory section.

GC Extractables/HPLC:	CHI-22-17-034
GC Volatiles:	CHI-22-19-003
GC/MS Volatiles and Semivolatiles:	CHI-22-20-038
Metals:	CHI-22-14-004, CHI-22-14-005, CHI-22-14-006
Wet Chemistry:	CHI-22-12-014

Primary Review

The primary review is often referred to as a "bench-level" review. In most cases, the analyst who generates the data (e.g., logs in, prepares and/or analyzes the samples) is the primary reviewer. In some cases, an analyst may be reducing data for samples run by an auto-sampler set up by a different analyst. In this case, the identity of both the analyst and the primary reviewer is identified in the raw data.

One of the most important aspects of primary review is to make sure that the test instructions are clear, and that all project specific requirements have been understood and followed.

Once an analysis is complete, the primary reviewer ensures, where applicable, that:

- Sample preparation information is complete, accurate, and documented.
- Calculations have been performed correctly.
- Quantitation has been performed accurately.
- Qualitative identifications are accurate.
- Manual integrations are appropriate.
- Data flags to indicate manual integrations are recorded.
- Manual integrations are authorized by a date and signature or initials of primary analyst.
- Client specific requirements have been followed.
- Method and process SOPs have been followed.
- Method QC criteria have been met.
- QC samples are within established limits.
- Dilution factors are correctly recorded and applied.
- Non-conformances and/or anomalous data have been properly documented and appropriately communicated.
- COC procedures have been followed.
- Primary review is documented by date and initials/signature of primary analyst.

Any anomalous results and/or non-conformances noted during the Primary Review are documented on the Data Review Checklist and on an SDR; and are communicated to the Section Manager and the Project Manager for resolution. Resolution can require sample reanalysis, or it may require that data be reported with a qualification. Non-conformances are documented per Section 4.9.

Secondary Review
The secondary review is also a complete technical review of a data and is performed by the Section Manager, analyst or data specialist. The secondary review is documented on the same Data Review Checklist as the primary review.

The following items are reviewed:
- Qualitative Identification
- Quantitative Accuracy
- Calibration
- QC Samples
- Method QC Criteria
- Adherence to method and process SOPs
- Accuracy of Final Client Reporting Forms
- Manual Integrations – Minimal requirement is to spot-check raw data files for manual integration, as verified by date and initials or signature of secondary data reviewer. Some regulatory programs require 100% secondary review of manual integrations.
- Completeness
- Special Requirements/Instructions

If problems are found during the secondary review, the reviewer must work with the appropriate personnel to resolve them. If changes are made to the data, such as alternate qualitative identifications, identifications of additional target analytes, re-quantitation, or re-integration, the secondary reviewer must contact the laboratory analyst and/or primary reviewer of the data so that the primary analyst and/or reviewer is aware of the appropriate reporting procedures.

Completeness Review

The completeness review includes the generation of a project narrative and/or cover letter which outlines anomalous data and non-compliances using project narrative notes and SDRs or CARs (non-compliance reports) generated during the primary and secondary review. The completeness review addresses the following items:

- Is the project report complete?
- Does the data meet with the client's expectations?
- Were the data quality objectives of the project met?

Are QC outages and/or non-conformances approved and appropriately explained in the narrative notes?

The laboratory Section Manager(s), Data Management personnel and the Project Manager contribute to the completeness review.

5.3.7 Data Integrity and Security

This section details those procedures that are relevant to computer systems that collect, analyze, and process raw instrumental data, and those that manage and report data.

Security and Traceability

Access to the laboratory's LabNet system, STL's proprietary LIMS, that collects, analyzes, and processes raw instrumental data, and those that manage and report data is both controlled and recorded. System users are granted access levels that are commensurate with their training and responsibilities.

Control of the system is accomplished through limitation of access to the system by users with the education, training and experience to perform the task knowledgeably and accurately. System users are granted privileges that are commensurate with their experience and responsibilities.

Computer access is tracked by using unique login names and passwords for all employees that have access to the computer system. Entries and changes are documented with the identity of the individual making the entry, and the time and date. Where a computer system is processing raw instrumental data, the instrument identification number as described in Section 5.4.1 is recorded. The system has the capability of maintaining audit trails to track entries and changes to the data. This function is activated on any computer system that has that capability (e.g., Target).

Verification
All the LabNet software programs have been verified prior to use and prior to the implementation of any version upgrades. Verification involves assessing whether the computer system accurately performs its intended function. Verification generally is accomplished by comparing the output of the program with the output of the raw data manually processed, or processed by the software being replaced. The verification of LabNet software programs are conducted by the QA manager with the assistance of the section managers and unit leaders. The QA manager documents the approval of the program verifications. All records of the verification are retained as QC records.

Validation
Software validation involves documentation of specifications and coding as well as verification of results. Software validation is performed by the QA manager on all in house programs. (LabNet) Records of validation include original specifications, identity of code, printout of code, software name, software version, name of individual writing the code, comparison of program output with specifications, and verification records as specified above. Records of validation are retained as QC records.

The QA manager must retain documentation of the validation process as defined above. The QA manager is the sole LabNet Methods Administrator at the laboratory and has the responsibility to validate any LabNet methods, calculations or criteria codes prior to use for sample analysis.

Auditing
STLs LabNet System Managers continually review the control, security, and tracking of IT systems and software.

Version Control
The laboratory maintains copies of outdated versions of software and associated manuals for all software in use at the laboratory for a period of 5 years from its retirement date. The associated hardware, required to operate the software, is also retained for the same time period.

5.4 Equipment

5.4.1 Equipment Operation

STL is committed to routinely updating and automating instrumentation. The laboratory maintains state of the art instrumentation to perform the analyses within the QC specifications of the test methods. The laboratory maintains an Equipment Tracking Form (CHI-22-09-068) for each piece of equipment and instrumentation that documents the following information:

- Identity
- Date In Service
- Manufacturer's Name, Model Number, Serial Number
- Current Location
- Preventative Maintenance Schedule

All equipment is subject to rigorous checks upon its receipt, upgrade, or modification to establish that the equipment meets with the selectivity, accuracy, and precision required by the test method for which it is to be used. All manufacturer's operations and maintenance manuals are kept up to date and accessible for the use of the equipment operator. Documentation of equipment usage is maintained using analytical run and maintenance logbooks.

5.4.2 Equipment Maintenance

STL employs a system of preventative maintenance in order to ensure system up time, minimize corrective maintenance costs and ensure data validity. All routine maintenance is performed as recommended by the manufacturer and may be performed by an analyst, instrument specialist or outside technician. Maintenance logbooks are kept on all major pieces of equipment in which both routine and non-routine maintenance is recorded.

Any item of equipment or instrumentation that has been subjected to overloading or mishandling, provides suspected results, has been shown by verification or otherwise to be defective, is new or not been used for an extended period of time, is taken out of services and tagged as "DO NOT USE INSTRUMENT". The tag is signed/dated by the person removing the item from service and noted as to the reason of in-operation (*Instrument and Equipment Out-of-Service Tagging*; UQA-012).

Any instrumentation that is brought back on-line must have MDLs and DOCs performed and have acceptance within prescribe criteria; or calibrated by a certified agency (e.g., balances or Class S weights) and tagged as being within calibration specifications; and proven to provide consistent measurements (e.g., refrigerators, eppendorf pipettes, ovens).

The return to analytical control following instrument repair is documented in the maintenance logbook. Maintenance logbooks are retained as QC records. Notation of the date and maintenance activity is recorded each time service procedures are performed. Maintenance logbooks are retained as QA records.

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Hewlett Packard GC/MS	Ion gauge tube degassing Pump oil-level check Pump oil changing Analyzer bake-out Analyzer cleaning Resolution adjustment COMPUTER SYSTEM AND PRINTER: Air filter cleaning Change data system air filter Printer head carriage lubrication Paper sprocket cleaning Drive belt lubrication	As required Monthly Annually As required As required As required As required As required As required As required As required
Gas Chromatograph	Compare standard response to previous day or since last initial calibration Check carrier gas flow rate in column Check temp. of detector, inlet, column oven Septum replacement Check system for gas leaks with SNOOP Check for loose/frayed wires and insulation Bake injector/column Change/remove sections of guard column Replace connectors/liners Change/replace column(s)	Daily Daily via use of known compound retention Daily As required W/cylinder change as required Monthly As Required As Required As Required As Required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63) Detector cleaning	Semi-annually As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Photoionization Detector (PID)	Change O-rings Clean lamp window	As required As required
HPLC	Change guard columns Change lamps Change pump seals Replace tubing Change fuses in power supply Filter all samples Change autosampler rotor/stator	As required As required Semi-annually or as required As required As required Daily As required
Balances	Class "S" traceable weight check Clean pan and check if level Field service	Daily, when used Daily At least Annually
Conductivity Meter	0.01 M KCl calibration Conductivity cell cleaning	Daily As required
Turbidimeter	Check light bulb	Daily, when used

Maintenance contracts are held on specific pieces of equipment where outside service is efficient, cost-effective, and necessary for effective operation of the laboratory. Table 9 lists STL's major equipment and the suggested maintenance procedures.

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
AA (Graphite Furnace)	Clean lens and furnace head	Daily
	Replace windows	As required
	Check or change cuvette	Daily
	Check & drain compressor drain	Daily
	Clean atomizer cell/furnace hood	Daily
	Nebulizer cleaned/dried	Weekly or as required
	Check/change marble stones	Weekly
	Clean filters	Weekly
	Change graphite tube/platform	As required
	Empty waste container	Daily
	Remove carbon tube and check wear	Daily
	Check sample introduction probe	Daily
Leeman Mercury Analyzer	Check tubing for wear	Daily
	Fill rinse tank with 10% HCl	Daily
	Insert clean drying tube filled with Magnesium Perchlorate	Daily
	Fill reductant bottle with 10% Stannous Chloride	Daily
ICP	Check pump tubing	Daily
	Check liquid argon supply	Daily
	Check fluid level in waste container	Daily
	Check filters	Weekly
	Clean or replace filters	As required
	Check torch	Daily
	Check sample spray chamber for debris	Monthly
	Clean and align nebulizer	Monthly
	Check entrance slit for debris	Monthly
	Change printer ribbon	As required
	Replace pump tubing	As required
UV-Vis Spectrophotometer	Clean ambient flow cell	As required
	Precision check/alignment of flow cell	As required
	Wavelength verification check	Semi-annually
Auto Analyzers	Clean sampler	Daily
	Check all tubing	Daily
	Clean inside of colorimeter	Daily
	Clean pump well and pump rollers	Quarterly
	Clean wash fluid receptacle	Weekly
	Oil rollers/chains/side rails	Weekly
	Clean optics and cells	Quarterly

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Deionized/Distilled Water	Check conductivity Check deionizer light Monitor for VOA's System cleaning Replace cartridge & large mixed bed resins	Daily Daily Daily As required As required
Drying Ovens	Temperature monitoring Temperature adjustments	Daily As required
Refrigerators/ Freezers	Temperature monitoring Temperature adjustment Defrosting/cleaning	Daily As required As required
Vacuum Pumps/ Air Compressor	Drained Belts checked Lubricated	Weekly Monthly Semi-annually
pH/Specific Ion Meter	Calibration/check slope Clean electrode	Daily As required
BOD Incubator	Temperature monitoring Coil and incubator cleaning	Daily Monthly
Centrifuge	Check brushes and bearings	Every 6 months or as needed
Water baths	Temperature monitoring Water replaced	Daily Monthly or as needed

5.4.3 Equipment Verification and Calibration

All equipment is calibrated prior to use (Initial Calibration) to establish its ability to meet the QC guidelines contained in the test method for which the instrumentation is to be used. All sample measurements are made within the calibrated range of the instrument and in compliance with method requirements. The calibration data, which includes instrument conditions and standard concentrations, is documented in pre-formatted instrument runlogs or within LabNet itself. The preparation of all reference materials used for calibration is documented via LabNet.

Once an instrument is calibrated, ongoing instrument calibration is demonstrated (Continuing Calibration) at the appropriate frequency as defined in the test method. Refer to the STL Corporate Policy *Selection of Calibration Points* (P-T-001), for guidance on using calibration data. Any instrument that is deemed to be malfunctioning is clearly marked and taken out of service. When the instrument is brought back into control, acceptable performance is documented.

5.4.3.1 Instrument Calibration

Specific instrument calibration procedures for various instruments are summarized further in this section, and detailed in the respective analytical methods. Typically, more than one analytical method is available for an analysis. These various methods and other program requirements (e.g., U.S. EPA CLP, AFCEE, NFESC, USACE, QAPPs, contracts, etc..) may specify different calibration requirements. Therefore, calibration details as specified in the respective laboratory SOPs, Technical Profiles, QAPP, program requirements, and contracts supersede the general instrument calibration procedures are described further in Table 10. Complete details are provided in each method SOP.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
Metals (ICAP)	Initial Calibration	Following a period of time sufficient to warm up the instrument, the ICP is calibrated prior to each analytical run or minimally every 24 hours. Calibration standards are prepared from reliable reference materials and contain all metals for which analyses are being conducted. Working calibration standards are prepared fresh daily. Quarterly, multi-concentration calibration is performed to document linearity. On a day-to-day basis, 4 calibration standards (blank, high standard, 50% standard, and 20% standard) are analyzed. Prior to an analytical run, the instrument is calibrated using three standards. An Initial Calibration Verification (ICV) standard is analyzed immediately after standardization, followed by an Initial Calibration Blank (ICB). The ICV is from a source other than that used for initial calibration and the ICB must be free of target analytes at and above the value to be reported or appropriate corrective action must be taken. ICP Interference Check Samples (ICSA/ICSAB) are analyzed at the frequency described in each method SOP.
	Continuing Calibration	The initial calibration is verified during the analysis sequence by analysis of a Continuing Calibration Verification (CCV) standard and a Continuing Calibration Blank (CCB). The response of the CCV must be within the SOP-specified criteria (e.g., $\pm$ 10% recovery of the true value). The CCB must be free of target analytes at or above the value to be reported or appropriate corrective action must be taken. If any ICVs/CCVs or blanks exceed their acceptance criteria, appropriate corrective action must be taken.
Atomic Absorption (GFAA/ CVAA)	Initial Calibration	Initial calibration will include analysis of a calibration blank and a minimum of four (4) calibration standards covering the anticipated range of measurement. Duplicate injections are made for each concentration. Response readings, e.g., absorbance, are recorded and the resultant standard calibration curve calculated. If the SOP or program-specified criteria are not met, appropriate corrective action must be taken. An ICV standard will be analyzed immediately after standardization. The ICV must be within SOP-specified criteria (e.g., $\pm$5% of the true value for drinking water, and $\pm$10% in most other cases), or the initial calibration must be repeated. The ICV must be from a source other than that used for initial calibration. An ICB will be analyzed after the ICV. The ICB must be free of target analytes at and above a concentration in which sample results are reported, or corrective action must be taken.
	Continuing Calibration	The initial calibration is verified during the analysis sequence by evaluation of a CCV standard and a CCB, as described above. The CCV value must be within SOP-specified criteria (e.g., $\pm$10% recovery of the true value except for mercury within $\pm$20 % of the true value). The CCB must be free of target analytes at and above the concentration reported in samples. If any ICVs/CCVs or blanks exceed their acceptance criteria, corrective action must be taken.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
Inorganic Colorimetric Methods	Initial Calibration	A full initial standard calibration curve will be prepared for all colorimetric analyses on a daily basis. Working standards to define this curve will include a minimum of five (5) concentrations which cover the anticipated range of measurement, plus a calibration blank. At least one of the calibration standards will be at a concentration which will enable verification of instrument response near the reporting limit as defined in Section 8.6 or a level suitable for meeting specific program requirements. The requirement for an acceptable initial calibration is described in the analytical SOP. If the criteria are not met, appropriate corrective action must be taken. Calibration data, e.g., correlation coefficient, is entered into the laboratory notebook, or associated instrument printouts, and retained with the sample data. In lieu of a full initial curve, a daily calibration verification may be analyzed. This daily calibration will at a minimum consist of a blank and a mid-range standard. Results must be within SOP-specified criteria. If not, reanalysis of the standards may be done once to verify the readings; otherwise, a new curve will be developed. For procedures that require pretreatment steps, a minimum of one standard shall be prepared with the pretreatment. If the pre-treated standard is within SOP-specified criteria, the curve will be used. If the pre-treated sample is not within the criteria, the reason will be determined. If it is determined that the difference between the curves is inherent in the procedure, the curve will be based on the standards prepared and carried through the pretreatment. An ICV will be analyzed immediately after the standardization, followed by an ICB. The ICV must be from a source other than that used for initial calibration. The ICV must be within SOP-specified criteria and the ICB must be free of target analytes or appropriate corrective action must be taken.
	Continuing Calibration	The initial calibration is verified during the analysis sequence by analysis of a CCB and a CCV. If any ICVs/CCVs or blanks exceed their acceptance criteria, analysis is terminated, and the instrument is recalibrated. All samples since the last valid calibration verification are reanalyzed.
Ion Chromatography	Initial Calibration	The ion chromatograph will be calibrated prior to each day of use. Calibration standards will be prepared from appropriate reference materials and will include a blank and a minimum of three concentrations to cover the anticipated range of measurements. At least one of the calibration standards will be at a concentration which will enable verification of instrument response near the reporting limit. If SOP-specified calibration criteria cannot be achieved, appropriate corrective action must be taken. Calibration data, e.g., correlation coefficient, will be archived with sample raw data.
	Continuing Calibration	A continuing calibration standard and blank will be analyzed at a frequency of 10% and at the end of the analysis shift. The response calculated as a percent recovery of the standard must meet SOP or program-specific criteria. The response of the blank must be less than the concentration to be reported for samples analyzed.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
GC/MS		All GC/MS instrumentation is calibrated to set specifications prior to sample analysis. These specifications vary depending on the requirements of the analytical program and the designated analytical method.
	Tuning and Mass Calibration	Mass spectrometers are calibrated with perfluorotributylamine (FC-43) or perfluorophenanthrene (FC- 5311) as required to ensure correct mass assignment. In addition, at the beginning of the daily work shift, the GC/MS system must be tuned with decafluorotriphenylphosphine (DFTPP) for semivolatiles analysis and 4-bromofluorobenzene (BFB) for volatiles analysis, and calibrated to target compounds. The majority of the laboratory work utilizes U.S. EPA-CLP or SW-846 protocols, which define the work shift as a 12-hour period initiated by the injection of DFTPP, BFB, or the dioxin/furan window mix. For drinking water programs (500 series methods), a 12-hour work shift is specified in the method for calibration frequency. For wastewater programs (600 series methods), the tune expires when the day's analytical sequence is complete; however, no time limit is given for the length of the daily GC/MS work shift. Ion abundances will be within the windows dictated by the specific program requirements.
	Initial Calibration	After an instrument has been tuned, initial calibration curves (minimum of 3-5 points) are generated for the compounds of interest. The low level standard must be at a concentration which will enable verification of instrument response near the reporting limit or at a concentration acceptable to meet program requirements. The other standards must extend through the linear working range of the detector. The parameters requiring quantitation must meet SOP or program-specified criteria prior to initiation of sample analysis. Any sample extracts containing parameters of interest which exceed the concentration of the high level standard, must be diluted to bring the parameters within the range of the standards. Instrument response to these target compounds are evaluated against SOP-specified criteria. Linearity is verified by evaluating the response factors (RF) for the initial calibration standards against SOP-specified criteria. Once an acceptable calibration is obtained, samples may be analyzed up until the expiration of the tune. At that time, the instrument must be re-tuned prior to further analysis. After acceptable tuning, a continuing calibration standard may be analyzed in lieu of a full multi-point calibration if the SOP-specified criteria are met. The majority of compounds analyzed for GC/MS comprise EPA's Target Compound List (TCL) or Priority Pollutant List (PPL). For add-on compounds not on the current TCL or PPL, initial calibration may be performed using a single point calibration of the additional compound(s), unless prior arrangements are made for a full three-to-five point calibration. Calibration data, to include linearity verification, will be maintained in the laboratory's records of instrument calibrations.
	Continuing Calibration	During each operating shift, a single calibration standard may be analyzed to verify that the instrument responses are still within the initial calibration determinations, as defined in the specific SOPs. If criteria cannot be met, appropriate corrective action must be taken.

5.5 Measurement Traceability

5.5.1 General

Traceability of measurements is assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard is subject to ongoing certifications of accuracy.

At a minimum, these include procedures for checking specifications for balances, thermometers, temperature, De-ionized (DI) and Reverse Osmosis (RO) water systems, automatic/eppendorf pipettes and other volumetric measuring devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards [with the exception of class A glassware (including glass microliter syringes that have a certificate of accuracy)].

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balances are calibrated on each day of use (*Balance Calibration, Care and Use*; UQA-003). All thermometers and temperature monitoring devices are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use (*Thermometer Calibrations*; UQA-034).

Laboratory DI and RO water systems have documented preventative maintenance schedules and the conductivity of the water is recorded on each day of use (*Water Quality*; UQA-035).

5.5.2 Reference Standards

The receipt of all reference standards is documented in LabNet. Standards are obtained from commercial vendors and sources may vary depending upon the availability of mixes and solutions from vendors. Each production unit is responsible to ensure, when available, that all standards are traceable to EPA, NIST, A2LA, SARMs and are accompanied by a Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis.

The receipt of each dry chemical, purchased stock solution or reference material to be used as a standard is assigned a unique ID number. The chemical name, manufacturer, lot number, date received, expiration date, date opened and initials of the analyst who opened the chemical are documented. The expiration dates for ampulated solutions shall not exceed the manufacturer's expiration date. Expiration dates for laboratory-prepared stock and diluted standards shall be no later than the expiration date of the stock solution or material or the date calculated from the holding time allowed by the applicable analytical method, whichever comes first. Expiration dates for pure chemicals shall be established by the laboratory and be based on chemical stability, possibility of contamination, and environmental and storage conditions. Expired standard materials shall be either revalidated prior to use or discarded. Revalidation may be performed through assignment of a true value and error window statistically derived from replicate analyses of the material as compared to an unexpired standard. The laboratory labels all standard and QC materials with expiration dates.

The preparation of all daughter solutions, whether a single or multiple-component stock, intermediate, or working standard solution, is documented in a standard solution preparation logbook, in a designated section of the analytical logbook or in the LabNet systems reagent program. This documentation references the Standard ID of the respective parent solution(s) used in its preparation, providing a solid trail back to the solution or chemical received from the vendor. These records include the standard name, final volume, matrix, final concentration, analyst initials, prep date and expiration date. A daughter solution should not have an expiration date which post-dates any of the parent solutions used in its preparation.

References standards are labeled with a unique Standard Identification Number, date received, and the expiration date. All documentation received with the reference standard or documentation of standard purity is retained as a QC record and references the Standard Identification Number. All efforts are made to purchase standards that are $\geq$ 97.0% purity. If this is not possible, the purity is used in performing standards calculations.

The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a different lot is acceptable for use as a second source. The appropriate QC criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an ICV or LCS is used as the second source confirmation.

Storage conditions, such as shelf life, ambient or chilled, controlled or restricted access, wet or desiccated, etc.., are in conformance with the specifications set in the associated method, the program requirements, or the manufacturer's recommendation, as appropriate.

5.5.3 Reagents

Reagents are, in general, required to be analytical reagent grade unless otherwise specified in method SOPs. Reagents must be, at a minimum, the purity required in the test method. The date of reagent receipt, date the reagent was opened, and the date of reagent preparation (where applicable) are documented in LabNet for reagent traceability.

5.6 Sampling

Sample representativeness and integrity are the foundations upon which meaningful analytical results rely. Where documented and approved SAPs and/or QAPPs are in place, they must be made available to the laboratory before sample receipt, and approved by laboratory management before sample receipt.

5.7 Sample Handling, Transport, and Storage

5.7.1 General

COC can be established either when bottles are sent to the field, or at the time of sampling. STL can provide all of the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory. Complete details for sample container preparation are contained within UCM-001. A summary of sample receipt is as follows with complete details available within the *Sample Receipt and Handling* SOP (USR-001).

Samples are received at the laboratory by the designated sample custodians and a unique LabNet job (batch) number and unique bottle ID is assigned. The following information is recorded for each sample shipment:

- Client/Project Name.
- Date and Time of Laboratory Receipt.
- Laboratory Job Number
- Signature or initials of the personnel receiving the cooler and making the entries.

Upon inspection of the cooler and custody seals, the sample custodian opens and inspects the contents of the cooler, and records the cooler temperature. If the cooler arrival temperature exceeds the required or method specified temperature range by $\pm 2^{\circ}C$ (for samples with a temperature requirement of $4^{\circ}C$, a cooler temperature of just above the water freezing temperature to $6^{\circ}C$ is acceptable); sample receipt is considered "compromised" and the procedure described in Section 4.7.1 is followed. All documents are immediately inspected to assure agreement between the test samples received and the COC.

Any non-conformance, irregularity, or compromised sample receipt as described in Section 4.7.1 is documented in an SDR and Sample Receipt Checklist and brought to the immediate attention of the Project Manager for resolution with the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the permanent project record.

Samples that are being tested at another STL facility or by an external subcontractor are repackaged, iced, and sent out under COC.

Following sample labeling as described in Section 5.7.2, the sample is placed in storage. Refrigerated storage coolers are maintained at $4 \pm 2^{\circ}C$. The temperature is continually being monitored by an electronic monitoring software program. *(Thermometer Calibrations and Electronic Monitoring: UQA-034)* All samples are stored according to the requirements outlined in the test method, and in a manner such that they are not subject to cross contamination or contamination from their environment.

Access to the laboratory is restricted to laboratory personnel or escorted guests as described in Section 5.2. Therefore, once sample possession is relinquished to the laboratory, the sample is in a designated secure area (e.g., the laboratory facility) accessible only to authorized personnel. Locked storage coolers are available for protocol (e.g., AFCEE and CLP) that require internal COC procedures.

5.7.2 Sample Identification and Traceability

The sample custodian organizes the sample containers, COCs, and all pertinent information associated with the samples. The sample identity is verified against all associated sample information. Any inconsistencies are documented via an SDR and forwarded to the Project Manager for resolution with the client prior to identifying the sample(s) into LabNet.

Each sample container is assigned a unique Sample Identification Number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label.

All unused portions of samples, including empty sample containers, are returned to the secure sample control area.

5.7.3 Sub-Sampling

Taking a representative sub-sample from a container containing a soil or solid matrix is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation.

After thoroughly mixing the sample within the sample container or transfer to a wip bag (or other suitable plastic bag), a sub-sample from various quadrants and depths of the sample are taken to acquire the required sample weight. Any non-homogenous looking material is avoided and noted as such within the sample preparation record.

5.7.4 Sample Preparation

Sample preparation procedures vary for each matrix and analytical method are as referenced in the laboratory SOPs.

5.7.5 Sample Disposal

Samples are retained in STL storage facilities for 30 days after the project report is sent unless prior written arrangements have been made with the client. Samples may be held longer or returned to the client per written request. Unused portions of samples are disposed of in accordance with federal, state and local regulations. The laboratory removes or defaces sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). Complete details on the disposal of samples, digestates, and extracts is available within the *Laboratory Waste Disposal Procedures* SOP (UWM-001).

5.8 Assuring the Quality of Test Results

5.8.1 Proficiency Testing

The laboratory analyzes Proficiency Test (PT) samples as required for accreditation and as outlined in NELAC. The laboratory participates in the PT program semi-annually for each PT field of testing for which it is accredited, according to the NELAC PT field of testing published guidelines. This includes drinking water, wastewater and solid/soil matrices.

The laboratory also participate various client PT programs, when submitted.

PT samples are handled and tested in the same manner (procedural, equipment, staff) as environmental samples. Results of PT samples are distributed to the laboratory section managers for review and corrective action, if required. Any required corrective action response to deficiencies is submitted to the QA department for review and are filed with the PT study records. PT test sample data is archived using the requirements for project and raw data record retention. Refer to the SOP: *PT Sample Tracking/Analysis (UQA-018)* for further details.

5.8.1.1 Double Blind Performance Evaluation

The laboratory participates in an annual double blind performance evaluation study. An external vendor is contracted to submit double blind samples to the laboratory. Both the level of customer service and the accuracy of the test results are assessed objectively by the external contractor, who provides a detailed report to the Corporate Quality Director and to the laboratory. This is administered as a double blind program in order to assess all facets of the laboratory's operations.

5.8.2 Control Samples

Control samples (e.g., QC indicators) are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity, selectivity, and interferences. Control samples must be uniquely identified and correlated to unique batches. Control samples further evaluate data based upon (1) Method Performance, which entails both the preparation and measurement steps; and (2) Matrix Effects, which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch.

Control sample types and typical frequency of their application are outlined Sections 5.8.2.1 through 5.8.2.5 and Tables 11 through 15. Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method and regulatory program control samples are as listed in Sections 7 and 8 of each method SOP.

5.8.2.1 Method Performance Control Samples: Preparation Batch

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment.

Control samples are added to each prep batch to monitor method performance (Table 11) and are processed through the entire analytical procedure with investigative/field samples.

Field blanks, equipment blank and trip blanks, when received, are analyzed in the same manner as other field samples. However, a field blank should not be selected for matrix QC, as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB".

5.8.2.2 Method Performance Control Samples: Matrix

Matrix control samples include sample duplicates (MD), sample matrix spikes (MS), and sample surrogate spikes. These control samples help monitor for potential physical and chemical effects which may interfere with the precision and/or accuracy of the selected analytical method. Since interferences can enhance or mask the presence of target analytes, matrix control samples measure the degree of interference and are used to assist in the interpretation of the analytical results. The laboratory avoids performing matrix QC on known field blank samples, such as trip blanks and rinsates, since these samples are not indicative of the sample matrix.

Table 11. Preparation Batch Control Samples

Control Type	Details	
Method Blank (MB)	Use	Monitors for potential contamination introduced during the sample preparation and analytical processes.
	Typical Frequency [1]	1 per batch of ≤ 20 samples per matrix type per sample extraction or preparation method.
	Description	Organics: Laboratory pure water for water samples or a purified solid matrix for soil or solid samples (when available or when requested); solid matrices commonly include sodium sulfate, vendor or agency supplied soil or solid, or purchased sand; these solids may require purification at the laboratory prior to use. Inorganics: Laboratory pure water for both water and soil or sediment samples. Volume/weights are selected to approximately equal the typical sample volume/weight used in sample preparation; and final results in a soil/solid batch may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison to actual field samples.
Laboratory Control Sample (LCS)	Use	Measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects.
	Typical Frequency [1]	1 per batch of ≤ 20 samples per matrix type per sample extraction or preparation method. For multi-analyte methods, the LCS may consist of surrogates in the blank matrix, and or a representative selection of target analytes/internal standards.
	Description	Prepared from a reference source of known concentration and processed through the preparation and analysis steps concurrently with the field samples. Aqueous LCS's may be processed for solid matrices unless a solid LCS is requested; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.
Known QC Sample	Use	Comply with regulatory requirements; check the accuracy of an analytical procedure; troubleshoot method performance problems; verify an analyst in training's ability to accurately perform a method; to verify the return-to-control after method performance problems; and may also be used as an LCS.
	Typical Frequency [1]	As defined by the client or QAPP.
	Description	Obtained from outside suppliers or agencies; generally require preparation from concentrated materials by dilution into a standard matrix; contain known analytes or compounds; acceptance limits are provided by the vendor.

[1] Denotes an STL required frequency.

Table 12. Matrix Control Samples

Control Type	Details	
Matrix Duplicate (MD)	Use	Monitors the effect of site matrix on the precision of the method; and of the reproducibility of laboratory preparation and measurement techniques. **Note:** Precision may also be affected by the degree of homogeneity of the sample, particularly in the case of non-aqueous samples or aqueous samples with particulates. Sample homogeneity and matrix effect should be considered when field samples are used to assess reproducibility. **Note:** A field duplicate, when received, measures Representativeness of sampling and the effect of the site matrix upon precision.
	Typical Frequency [1]	1 per 20 samples per matrix or per SAP/QAPP [2].
	Description	Performed by analyzing two aliquots of the same field sample independently; analyzed for each associated sample matrix (e.g., when requested by the client or the analytical method).
Matrix Spike (MS)	Use	Measures the effect of site sample matrix on the accuracy of the method.
	Typical Frequency [1]	1 per 20 samples per matrix or per SAP/QAPP.
	Description	Aliquot of a field sample which is spiked with the analytes or compounds of interest; analyzed for each associated sample matrix (when requested by the client or analytical method). The determination of MS percent recovery (% R) requires an analysis of a fortified sample and a non-fortified sample under the same procedural conditions (e.g., sample volumes, dilutions, procedural conditions, etc..). The concentration determined in the non-fortified sample is subtracted from the fortified sample concentration before determining the %R. The degree of homogeneity of the sample, particularly in the case on non-aqueous samples or samples with particulates, may affect the ability to obtain representative recoveries.
Matrix Spike Duplicate (MSD)	Use	Measures effect of site sample matrix on precision of method.
	Typical Frequency [1]	1 per 20 samples per matrix, when requested by the client or the analytical method, or per SAP/QAPP [2].
	Description	Alternative to sample duplicate. Generally, inorganic protocols specify an MD/MS and organic protocols specify an MS/MSD.
Surrogate Spike	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Every QC and analytical sample.
	Description	Compounds similar to the target analytes in structure, composition and chromatography, but not typically found in the environment, are added to each QC and analytical sample, prior to preparation (e.g., extraction). If the surrogates in an analytical batch do not all conform to established control limits, the pattern of conformance in investigative and control samples is examined to determine the presence of matrix interference or the need for corrective action.
Internal Standards	Use	Monitor the qualitative aspect of organic and inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] Denotes an STL required frequency.
[2] Either an MSD or an MD is required per 20 samples per matrix or per SAP/QAPP.

5.8.2.3 Matrix QC Frequencies

The frequency of matrix QC indicators depends on regulatory program compliance, a project's data quality objectives, or a client's requirements. The following frequency will be applied to samples when the regulatory programs are known and it does not conflict with project or client requirements.

Table 13. EPA Program Requirements

Program	Description [1]
SDWA	MD performed at a 10% frequency or 1 per preparation batch of ≤10 samples, whichever is more frequent.
CWA	MS (GC methods) and MD is performed at a 10% frequency or 1 per preparation batch of ≤10 samples, whichever is more frequent. For GC/MS Methods, MS is performed at a 5% frequency or 1 per preparation batch of ≤20 samples, whichever is more frequent.
RCRA	MS/MSD or MS/MD is performed at a rate of 5% per client (independent of the preparation batch). For clients submitting less than 10 samples, the method matrix QC requirement may be satisfied by another clients sample within the same prep batch unless the paperwork indicates a client requirement for matrix QC. Matrix QC will only be reported to the client who owns the data.
U.S. EPA CLP	MS/MSD or MS/MD is performed at a rate of 5% or 1 set per Sample Delivery Group (SDG) per matrix, independent of the prep batch. For NFESC samples, samples are processed in simultaneous or continuous batches.

[1] MS, MSD and MD may not be applicable to some analytical protocols because of the nature of the sample or protocol.

5.8.2.4 Method Performance Control Samples: Instrument Measurement

Control samples are used to ensure that optimum instrument performance is achieved. These samples help ensure that the proper identification and quantitation of target compounds or analytes are achieved. The instrument control samples appropriate to each analytical technique are described in laboratory SOPs for each respective method. A brief description of these checks is included in Table 14.

Table 14. Instrument Performance Control Samples

Control Type	Description	
Inorganics		
ICV	Use	Calibration standard of known concentration prepared from a source other than that used for the calibration standards.
	Sequence	Analyzed after the standard curve to confirm calibration.
ICB	Use	Blank water or solvent; confirms the calibration and assures that any potential contamination is less than the reporting limit.
	Sequence	Analyzed immediately after the ICV.
ICP Interference Check Samples (ICSA/ICSB)	Use	Verifies the absence of spectral interferences.
	Sequence	Analyzed consecutively at the beginning of each eight hour analytical sequence, after the ICV/ICB, and again at an eight hour frequency following a CCV/CCB. When CLP protocols are followed, the ICSA/B will be analyzed with the analytical sequence, before the final CCV/CCB.
Reporting Limit Verification Standard (CRA & CRI)	Use	Verifies linearity near the reporting limit for CLP metals analyses. (Note: CRI is at a level 2X the CRDL; CRA is near the CRDL).
	Sequence	Analyzed after the ICB. The CRI is also analyzed at the end of the eight hour analytical sequence, prior to analysis of the final CCV/CCB.
CCV	Use	Confirm that the instrument performance has not significantly changed during the analytical sequence; to verify stable calibration throughout the sequence; and/or to demonstrate that instrument response did not drift over a period of non-use. Made from a source other than that used for the standard curve.
	Sequence	Analyzed at 10% or every two hours, whichever is more frequent; also analyzed at the end of the analytical sequence.
CCB	Use	Water blank used to confirm that the baseline has not drifted and to monitor for contamination at the reporting limit.
	Sequence	Analyzed at a rate of 10% for inorganics and at a rate of 1 per 10 readings/injections or every two hours, whichever is more frequent, for CLP metals; also analyzed at the end of the analytical sequence.
ICP Metals Linear Range	Use	Verify linearity and document the upper limit of the calibration range for each element.
Analysis Standard (LRS)	Sequence	Performed quarterly with a blank and a minimum of five standard concentrations to cover the anticipated range of measurement; one of the calibration standards will be at or near the reporting limit. The calibration curve generated must have a correlation coefficient $\geq$ 0.995 in order to consider the responses linear over that range.
ICP Inter-Element Correction (IEC)	Use	Correction factors for spectral interference (particularly due to Al, Ca, Fe, and Mg).
	Sequence	Determined at least annually for all wavelengths used for each analyte reported by ICP; or any time the ICP is adjusted in any way that may affect the IECs.

Table 14. Instrument Performance Control Samples

Control Type		Description
		Organics
GC/MS Tuning & Performance	Use	Ensures correct mass assignment and is monitored through response to target compounds during initial and continuing calibration, with minimum response criteria for specified system performance check compounds (SPCCs), and linearity is verified by evaluating the response factors (RF) for calibration check compounds (CCCs).
	Sequence	Tuned at the beginning of the daily work shift. Throughout the analysis, blanks, internal standard areas, surrogates, chromatographic baseline, resolution of peaks, and overall quality of the chromatography are used collectively to monitor instrument performance.
GC & HPLC Instrument Performance	Use	Monitored through retention time shift evaluation, linearity checks, and degradation checks of selected target compounds (e.g., for Endrin or DDT as appropriate).
	Sequence	Continuing calibration verification (e.g., blanks, shifts in chromatographic baseline or retention times, resolution of peaks, and overall quality of the chromatography) throughout the analytical sequence is accomplished through analysis of calibration check standards.

5.8.2.5 Method Performance Control Samples: Analysis Batch

Matrix specific control samples are used to assess the precision and accuracy of the method as applied to the specific sample matrix. These indicators provide information on sample matrix effects that is independent of the efficiency of the preparatory technique. The method performance control samples appropriate to each analytical technique are identified in the respective method. A brief description of these checks is included in Table 15.

These control samples are performed to provide a tool for evaluating how well the method performed for the respective matrix. These values are used by the client to assess the validity of a reported result within the context of the project's data quality objectives. For matrix specific QC results falling outside laboratory control limits which are attributed to matrix affects, no systematic corrective action is taken.

Table 15. Analysis Batch Performance Control Samples

Control Sample Type	Description	
ICP Serial Dilution	Use	5X Dilution of a field sample (performed at the instrument) to check for possible physical and/or chemical interferences.
	Sequence	5% of field samples or 1 per ≤20 samples per batch.
GFAA Analytical Bench Spike	Use	Required by the method; prepared at the instrument by fortifying the digestate with a known quantity of the analyte of interest.
	Sequence	Performed on each sample immediately following the unspiked original analysis.
Method of Standard Addition (MSA)	Use	When specified by the analytical protocol or by client request.
	Sequence	When specified by the analytical protocol or by client request.

5.8.3 Statistical Control Limits and Charts

Statistical control limits and control charts are used to establish method performance of a given analysis and to monitor trends of QC results graphically over time. Once a data base of the laboratory results for a method/matrix/QC analyte combination is established, the acceptability of a given analysis of that QC parameter (and of the analytical batch to which it belongs) can be evaluated in light of the laboratory's normal performance. This is intended to help identify problems before they might affect data. Often, patterns of response that are not at all evident in sets of numbers are very distinct when the same values are viewed as a chronological graph.

Establishment of Limits
The purpose of using statistical control limits is to define, for each analyte in a given method/matrix/QC type combination, a range of expected values. This range encompasses the random variation that occurs normally in the laboratory and allows one to evaluate control samples in that context, rather than according to an arbitrary or external set of values. Limits for accuracy and precision are defined below:

Accuracy
As recoveries of a QC analyte in a given matrix are tabulated over time, a mean value for recovery is established, as is the standard deviation (s) of those recoveries. If the analysis is in statistical control (e.g., if the set of QC recoveries over time show random variation about the mean) approximately 99.7% of all recoveries for that QC will fall within three standard deviations (3s) of the mean. Thus, assuming that the mean itself is an acceptable level of recovery, the values corresponding to 3s above and 3s below the mean are defined as the Control Limits. Any single recovery outside these values is assumed to have resulted from some circumstance other than normal variation and shall be investigated.

Roughly 95% of points should fall within 2s of the mean. The values +2s and -2s are the Warning Limits. Any normal result has approximately a 1/20 chance of being between 2s and 3s from the mean, so a result in this region doesn't necessarily warrant corrective action, but attention should be paid to such points.

Precision
Precision is used to indicate matrix variability so that appropriate decisions can be made by the client when repeated analyses vary significantly. The coefficient of variation, expressed as a percentage (e.g., the %RSD) for the data set used to calculate accuracy control limits defines the control limit for precision. Duplicate analyses of the QC samples, such as duplicates or MS/MSD, should have an RPD less than or equal to this established precision control limit to be considered free of matrix interferences.

The laboratory calculates statistical control limits on an annual basis. Such limits are available on a project or QAPP-specific basis.

5.8.4 Calibration

Calibration protocols are method-specific, are briefly described in Table 10 and are defined in the Sections 6 & 7 of the method SOPs.

5.8.5 Glassware Cleaning

All glassware is thoroughly cleaned prior to use to ensure that sample integrity is not affected from artifacts caused by contaminated glassware.

A summary of general cleaning procedures follows with details provided in the *Laboratory Glassware Cleaning* SOP (UQA-009):

General laboratory glassware is cleaned with a low- or non-phosphate detergent, followed by thorough rinsing with tap water and deionized water.

Volumetric flasks and pipettes used for inorganics (method dependent), test tubes and caps used for micro-COD procedures, phosphate glassware, and metals-related glassware include an acid-washing step.

BOD glassware cleaning includes a nitric or sulfuric acid and/or a NOCHROMIX-washing step.

Organic glassware includes a solvent-wash.

Non-volumetric organic glassware may optionally be kiln dried at 400°C.

5.8.6 Permitting Departures from Documented Procedure

Where a departure from a documented SOP, test method, or policy is determined to be necessary, or unavoidable, the departure is documented in a CAR or SDR and reported in the case narrative. In most cases, these departures can be made with the approval of the section manager, project manager and the client. Issues of serious concern, as determined by the Section Manager or Project Manager, will be brought to the attention of the Laboratory Director and/or QA Manager. In some instances, it is appropriate to inform the client before permitting a departure. The Project Manager will make the determination as to the degree of notification required by the client.

On rare occasions, special analytical techniques will be requested for research, project specific requirements, or client needs. In these instances, SOPs may not be available, however, the analyst will thoroughly record the analytical steps and observations within a bound preformatted logbook.

5.8.7 Development of QC Criteria, Non-Specified in Method/Regulation

Where a method or regulation does not specify acceptance and/or rejection criteria, the laboratory must examine the data user's needs and the demonstrated sensitivity, accuracy and precision of the available test methods in determining appropriate QC criteria.

Data users often need the laboratory's best possible sensitivity, accuracy, and precision using a routinely offered test method, or are unsure of their objectives for the data. For routine test methods that are offered as part of STL's standard services, the laboratory bases the QC criteria on statistical information such as determination of sensitivity, historical accuracy and precision data, and method verification data. The method SOP includes QC criteria for ongoing demonstration that the established criteria are met (e.g., acceptable LCS accuracy ranges, precision requirements, method blank requirements, initial and continuing calibration criteria, etc..).

In some cases, a routine test method may be far more stringent than a specific data user's needs for a project. The laboratory may either use the routinely offered test method, or may opt to develop an alternate test method based on the data user's objectives for sensitivity, accuracy, and precision. In this case, it can be appropriate to base the QC criteria on the data user's objectives, and demonstrate through method verification and ongoing QC samples that these objectives are met.

For example, a client may require that the laboratory to test for a single analyte with specific DQOs for sensitivity, accuracy, and precision as follows: Reporting Limit of 10 ppm, Accuracy $\pm$25%, and RSD of <30%. The laboratory may opt to develop a method that meets these criteria and document through the Method Blank results, MDL study, and LCS results that the method satisfies those objectives. In this case, both the method and the embedded QC criteria have been based on the client's DQOs.

In some cases, the data user needs more stringent sensitivity, accuracy, and/or precision than the laboratory can provide using a routine test method. In this case, it is appropriate that the laboratory provide documentation of the sensitivity, accuracy, and precision obtainable to the data user and let the data user determine whether to use the best available method offered by the laboratory, or determine whether method development or further research is required.

5.9 Project Reports

The SOP for data package assembly and reporting formats is defined in the *Data Management, Process Operation SOP* (UDM-001) and a summary of this procedure follows.

Analytical reports comprise final results (uncorrected for blanks and recoveries unless specified), methods of analysis, levels of reporting, surrogate recovery data, and method blank data. In addition, special analytical problems will be noted in the case narratives. The number of significant figures reported are consistent with the limits of uncertainty inherent in the analytical method. Consequently, most analytical results will be reported to no more than two (2) or three (3) significant figures. Data are normally reported in units commonly used for the analyses performed.

Concentrations in liquids are expressed in terms of weight per unit volume (e.g., milligrams per liter, mg/L). Concentrations in solid or semi-solid matrices are expressed in terms of weight per unit weight of sample (e.g., micrograms per kilograms, ug/kg). Reporting limits take into account all appropriate concentration, dilution, and/or extraction factors, unless otherwise specified by program requirements (e.g., IRPMS reports).

A client report is generated with various steps of approval prior to printing of the final version. If any analytical anomalies were encountered during the analyses, e.g., an out-of-control matrix duplicate, it is documented in a case narrative. The case narrative is prepared by the respective operating unit and submitted to the data management section to insert in the final report.

The final report forms are printed, data packages are organized, a glossary of flags and acronyms is added, and reports are paginated.

5.9.1 General

The criteria described in Section 5.9.2 apply to all Project Reports that are generated under NELAC requirements. The criteria described in Section 5.9.3 and 5.9.4 apply to all Project Reports.

5.9.2 Project Report Content

- Title
- Laboratory name, address, telephone number, contact person
- Unique Laboratory Project Number
- Name and Address of Client
- Client Project Name (if applicable)
- Laboratory Sample Identification
- Client Sample Identification
- Matrix and/or Description of Sample
- Dates: Sample Receipt, Collection, Preparation and/or Analysis Date
- Definition of Data Qualifiers
- Reporting Units
- Test Methods
- Report Paginated

The following are required where applicable to the specific test method or matrix:

- Solid Samples: Indicate Dry or Wet Weight
- Whole Effluent Toxicity: Statistical package used
- If holding time $\leq$ 48 hours, Sample Collection, Preparation and/or Analysis Time
- Indication by flagging where results are reported below the quantitation limit.

5.9.3 Project Narrative

A Project Narrative and/or Cover Letter is included with each project report and, at a minimum, includes an explanation of any and all of the following occurrences:

- Non-conformances
- "Compromised" sample receipt (see Section 4.7.1)
- Method Deviations
- QC criteria failures

Project Release

The Project Manager or his designee authorizes the release of the project report with a signature.

Where amendments to project reports are required after issue, these are documented in the form of an RDR (refer to Section 4.8) and can be in the form of a separate document and/or electronic data deliverable resubmittal. The revised report is clearly identified as revised with the date of revision and the initials of the person making the revision. Specific pages of a project report may be revised using the above procedure with an accompanying cover letter indicating the page numbers of the project revised. The original version of the project report will be kept intact and the revisions and cover letter included in the project files.

5.9.4 Subcontractor Test Results

Subcontracted data is clearly identified as such, and the name, address, and telephone number for the laboratory performing the test is included in the project report. Subcontracted results from laboratories external to STL are not reported on STL report forms or STL letterhead. Test results from more than one STL facility are clearly identified with the name of the STL facility that performed the testing, address, and telephone number for that facility. Data from subcontractors' reports may be added to an STL electronic deliverable.

Data subcontracted within STL may be reported on the originating laboratory's report forms provided the following mandatory requirements are met:

- The name, address, and telephone number of the facility are provided.
- Analytical results produced by the STL intra-company subcontractor are clearly identified as being produced by the subcontractor facility.
- The intra-company subcontractor's original report, including the chain of custody is retained by the originating laboratory.
- Proof of certification is retained by the originating laboratory.
- All information as outlined in Section 5.9.2 is included in the final report where the report is required to be compliant with NELAC, for both the originating and subcontracting laboratory.

5.9.5 Electronic Data Deliverables

Electronic Data Deliverables (EDD) are routinely offered as part of STL's services. STL offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the EDD development staff by the PM for review and undergo the contract review process in Section 4.4.1. Once the laboratory has committed to providing diskettes in a specific format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained as a QC record.

EDDs are subject to a secondary review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory demonstrates that it can routinely generate that EDD without errors. Any revisions to the EDD format are reviewed until it is demonstrated that it can routinely be generated without errors. (*EDD SOP*: UIS-001)

5.9.6 Project Report Format

STL offers a wide range of project reporting formats, including EDDs, short report formats, and complete data deliverable packages modeled on the Contract Laboratory Protocol (CLP) guidelines. More information on the range of project reports available in the Data Management SOP (UDM-001). Regardless of the level of reporting, all projects undergo the levels of review as described in Section 5.3.6.

Appendix. List of Cited SOPs and Work Instructions

Cited Sec. No(s)	Description	Document No.
1.6; 5.7.1	Container Management: Process Operation	UCM-001
1.6; 4.4.2	Project Management: Project Planning Process	UPM-003
4.1	Signature Authority	UQA-030
4.1.1	Work Instruction: Equipment & Instrumentation Listing	CHI-22-09-103
4.1.2.9	Internet Use Policy Electronic Mail Use Computer System Account and Naming Policy Computer System Password Policy Software Licensing Policy Virus Protection Policy	P-I-001 P-I-002 P-I-003 P-I-004 P-I-005 P-I-006
4.3.1	Document Control	UQA-006
4.3.1.1; 5.3.2	Approved SOP Listing	CHI-22-09-SOP
4.3.2; 4.12.3	Data Management: Record Retention & Purging	UDM-002
4.4.2	Project Kick-Off Meetings	UPM-002
4.4.2	Production Meetings	UPM-004
4.4.3.6	IDL's for CLP Metals and Cyanide	UQA-010
4.4.3.6; 5.3.5	Method Detection Limits (MDLs)	UQA-017
4.5	Work Sharing Process - Policy	S-C-001
4.6	Procurement Quality Assurance Process	UQA-020
4.6.1	Testing Solvents and Acids	S-T-001
4.7.2	Client Confidentiality	UQA-004
4.8; 4.11	Sample Discrepancy Reports (SDRs) / Resubmitted Data Reports (RDRs) / Corrective Action Reports (CARs)	UQA-029
4.8; 4.11	Quality Systems Management Review	UQA-002
4.11	Preventive Action Measures	UQA-019
4.12.2	Work Instruction: Records Management Form	CHI-22-05-032
4.13	Internal Audits	UQA-013
5.1.2	Training Program: Mechanisms and Documentation Processes Defined by Operational Assessment	UQA-014
5.1.2	STL Chicago Demonstration of Capability Certification Statement	CHI-22-09-271
5.1.2	STL Chicago Yearly Method Capability Review Work Instruction: WC	CHI-22-09-279
5.1.3	Ethics Policy	P-L-006
5.3.1	Work Instruction: Methods Capabilities	CHI-22-09-255
5.3.2	SOP Change Protocol	UQA-032
5.3.5	MDL Policy	S-Q-003
5.3.6.1	Acceptable Manual Integration Practices	S-Q-004
5.3.6.2	Data Review Checklists GC Extractables / HPLC GC Volatiles GC/MS: Volatiles and Semivolatiles Metals Wet Chemistry	 CHI-22-17-034 CHI-22-19-003 CHI-22-20-038 CHI-22-14-004; 5; 6 CHI-22-12-014
5.4.1	Work Instruction: Equipment Tracking Form	CHI-22-09-068
5.4.2	Instrument and Equipment Out-of-Service Tagging.	UQA-012

Appendix. List of Cited SOPs and Work Instructions

Cited Sec. No(s)	Description	Document No.
5.4.3	Selection of Calibration Points	P-T-001
5.5.1	Balance Calibration, Care and Use	UQA-003
5.5.1; 5.7.1	Thermometer Calibrations and Electronic Monitoring	UQA-034
5.5.1	Water Quality	UQA-035
5.7.1	Sample Receipt: Handling and Processing	USR-001
5.7.5	Laboratory Waste Disposal Procedures	UWM-001
5.8.1	PT Sample Tracking/Analysis	UQA-018
5.8.5	Glassware Cleaning Procedures	UQA-009
5.9.5	EDD SOP	UIS-001
5.9; 5.9.6	Data Management: Process Operation	UDM-001

Laboratory Standard Operating Procedure

Gas Chromatography/Mass Spectrometer-Volatiles

SOP No.	Revision No.	Date	Page
UMV-OLC2.1	03	07/21/04	1 of 51

TITLE: GAS CHROMATOGRAPHY MASS SPEC - VOLATILES U.S. EPA CLP Document No. OLC02.1

Updated by:	Signature:	Date
JoAnn M. Petruszak Supervisor, GC/MS Volatiles	Jo M. Petruszak	07-19-04
Marilyn G. Krueding QA Specialist	Marilyn G. Krueding	07-16-04

Approved by:	Signature:	Date
Gary L. Rynkar Section Manager, GC/MS	Gary Rynkar	7/20/4
David W. Mazur Env. Health & Safety Coor.	D. W. Mazur	7/20/04
Terese A. Preston Quality Manager	Terese A. Preston	07/19/04

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Full Signature Approvals Are Kept on File
with STL's QA Standard Practice Records

1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the guidelines for the analysis of Volatile Organic Compounds (VOCs) by Gas Chromatography/Mass Spectrometry (GC/MS) by U.S. EPA CLP Document No. OLC02.1.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants". MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually.

1.1.2 Reporting Limits

Reporting limits are defined as Contract Required Quantitation Limits (CRQL) for CLP analyses. The Target Compound List (TCL) and their CRQLs are listed in Table 1. CRQL's are highly matrix dependent and will vary.

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Manual (LQM).

1.2 Summary of Method

This method is used to analyze samples containing low level volatile organic compounds. It is applicable to well/ground water and drinking water.

This method can be used to quantify most volatile organic compounds that have a boiling point <200°F. It is also limited to those compounds that elute as sharp peaks from a capillary column.

A portion of sample, measured into a 40 mL vial, is purged with an inert gas. The volatile compounds are transferred to a trap, containing retarding materials.

The trap is then backflushed with the inert gas and rapidly heated to effectively transfer the compounds to the GC column. The GC oven is temperature ramped to separate the compounds and introduce them to the source.

The mass filter separates the ions, which are then detected by the analyzer. The data system then provides qualitative and quantitative information concerning the sample.

Instrument calibration occurs every 12 hours, or prior to analysis. Instrument maintenance is performed, as needed, on a daily, monthly or yearly basis.

2.0 INTERFERENCES

- External interferences can be caused by contaminants from sample containers, preparative glassware and reagents, syringes and columns and manifest themselves as high background and/or discrete peaks. Some contaminants are also introduced through the sample vial seal and/or instrument sample connections. Proper glassware preparation, sample handling and instrument maintenance should eliminate these sources. A laboratory method blank is analyzed prior to any analysis to show absence of any contaminants. Reagent water sampled in the laboratory and carried through all field operations (Trip Blank) is also analyzed to show absence of contaminants from field sampling.

- Carryover is also another source of contamination. Any time a high level sample is analyzed, the next sample in the batch is checked for carryover. If carryover is suspected, that sample is re-analyzed. The position is rinsed with methanol/water. If carryover is excessive and continues into the next samples, the batch is aborted/paused, the column and trap baked, and/or blanks analyzed until all contamination is absent. If further response is required (i.e., trap replacement), it is documented in the maintenance logbook.

- Internal interferences can be purged from the sample with the target compounds and appear as elevated baselines or distinct peaks. Internal interferences most often manifest themselves as low/high recoveries of surrogate/matrix spike compounds. Matrix interferences vary from sample to sample.

- The volatile laboratory must be free of solvents and toluene. Common air-borne laboratory contaminants, Dichloromethane and Acetone, are allowed up to 3X the CRQL. The common contaminants 2-Butanone and Cyclohexane are also allowed up to 3X the CRQL.

3.0 SAFETY

- Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

3.1 Specific Safety Concerns or Requirements

- The GC contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.
- All employees will adhere to the practices and policies in the STL Corporate Safety Manual (CSM) and will read the MSDS's for the materials used in this method before handling or using the material.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: **This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

4.1 Current Hardware/Software

- 5 Hewlett-Packard 5890 GC interfaced with a 5972 MSD. Equipped with DB-624 column.
- 3 Hewlett-Packard 6890 GC interfaced with a 5973 MSD. Equipped with DB-624 column.
- 7 Tekmar 3000 concentrators, 1 PTS Enchon concentrator in connection to 8 Varian Archon Autosamplers for eight systems.
- 1 Combi PAL Static Headspace Screener in connection with Hewlett-Packard 5890 GC interfaced with a FID equipped with DB-624 column.
- 5-Hewlett-Packard Chemstations B.02.04 software and peripheral hardware.
- 1-Hewlett-Packard Chemserver 9000 series running HP-UX10.2 OS with Target 3.5.
- 1-Hewlett-Packard Chemstations G1701DA vD0.1 software and peripheral hardware.
- 1-Hewlett-Packard Chemstations G1701DA vD.0 software and peripheral hardware.
- 1-Hewlett-Packard Chemstations G1701CA vC.0 software and peripheral hardware.

Each temperature-programmable chromatograph is interfaced with a mass-selective detector (MSD) capable of scanning from 35 - 260 amu every second or less using 70 volts of electron energy in the electron ionization mode. The system is capable of producing an acceptable spectrum of bromofluorobenzene when 50 ng/5 mLs is purged.

4.2 Data System

The analytical systems are interfaced with stand alone PC's which are Pentium based systems running Agilent Chemstation. This system is capable of continuous acquisition and storage of mass spectral data. Completed data files are automatically transferred to the Chemserver Target 3.5 processing software which is capable of plotting specific masses versus time or scan numbers (Extracted Ion Current Profile) and integration of that abundance. The system also stores the data. The library NBS75K resides on the Chemserver.

4.3 Data File Name / Batch Directory Assignment

Each job # (assigned at log-in to a batch of samples) is assigned a code at the time that the first sample is analyzed. Tune, standard, blank, and lab control sample (LCS) data files are designated by specific letters unique to each instrument in conjunction with the appropriate month and day (Example: 3b0318 = instrument #3, first 12 hour BFB tune, March 18). During transfer of the files to the Chemserver, a unique batch directory is created on Target per instrument, date and tune.

4.4 Miscellaneous

- assorted syringes (10, 25, 50, 100, 500 and 1000 uL)
- 5 mL luer-lock gas-tight syringes
- top-loading balance, capable of weighing to ± 0.1 g, stainless steel spatula
- assorted amber and clear Teflon-lined screw-capped vials (1.5-2.0 mL, 3.5-5.0 mL)
- cleaned 40 mL vials w/Teflon-lined screw-caps
- assorted volumetrics (10 mL, 25 mL, 50 mL and 100 mL)

5.0 REAGENTS AND STANDARDS

All neat standards/kits received are entered into LabNet (and recorded in the Neat Standards Logbook). A code is written on the bottle/kit and entered into LabNet (and recorded in the logbook). All neat standards are then stored in a separate freezer at ~ –10 °C until needed. The standard is issued a unique ID# [i.e., Neat Standards Reference Number (NSRN)] which is used to track all standards as they are used as is or in preparation of stock/working solutions.

5.1 Reagents

5.1.1 Reagent Water

One (1) liter of Milli-Q water is continuously purged with pre-purified nitrogen. The reagent water is routinely demonstrated to be interference-free. All compounds are <CRQL or 3x CRQL for methylene chloride, cyclohexane, acetone and 2-butanone.

5.1.2 Methanol

All new lot numbers of P & T J.T.Baker Methanol are analyzed and verified to be free of contaminants. This information is available on the STL Oasis Web-Site that can be accessed by all analysts. The currently approved lot numbers are listed on this site. A copy of the section of the spreadsheet is posted in the GC/MS VOA Lab. Upon receipt of a case of methanol, the lot number is verified on the spreadsheet. If the lot number does not appear on the hard-copy, the analyst will go to the web-site and verify the analysis. A new spreadsheet with the most current lot number will be posted. In the case where the new lot number can not be verified electronically, the GC/MS lab will analyze a portion of the methanol, verify it to be free of contaminants, and record the analysis information in the Methanol Check Logbook.

5.2 Surrogate Spiking Solution

Stock surrogates are purchased as a neat solution from Ultra in a 1.5-2.0 mL ampule. The following surrogates are used:

Surrogate Spiking Mix	Concentration
4-Bromofluorobenzene	2500 ppm

NOTE: The surrogates 1,2-Dichloroethane-d4 and Toluene-d8 are also found in this mix but are not quantitated for by this method.

The transfer is entered into LabNet (and recorded in the Standard Preparation Log). The standard issued is another unique ID# [i.e., SRN (Standard Reference Number)] which can be traced back to the parent ID# (i.e., NSRN with the date of receipt, date of opening, and the supplier). Working surrogate solution is prepared with the internal standard solution (refer to Section 8.3.)

- Life of Standard: 1-year unopened; once opened, they are used for a period of 6 months or until used.
- Storage Requirements: Stored in a freezer at ~ -10°C in the dark and kept for a period of one year unopened.*

* If the stock solution has manufacturers' expiration date, that is assigned. If the date is not evident, one year is assigned to un-opened ampules. This is applicable for all "neat" standards.

5.3 Internal Standard Spiking Solution

Stock internal standards are purchased as a neat solution from Ultra in 1.5 -2.0 mL ampules. The following internal standards are used:

Internal Standard Spiking Mix	Concentration
1,4-Difluorobenzene	\
Chlorobenzene-d5	2000 ppm each
1,4-Dichlorobenzene-d4	/

NOTE: The internal standard Pentafluorobenzene is also included in this solution but is not used for quantification.

After opening, the remaining mixture is transferred to a 1.5 - 2.0 mL amber Teflon-lined screw-capped vial. The transfer is entered into LabNet (and recorded in the Standard Preparation Log). The standard issued is another unique ID# [i.e., SRN (Standard Reference Number)] which can be traced back to the parent ID# (i.e., NSRN with the date of receipt, date of opening, and the supplier).

- Life of Standard: 1-year unopened; once opened, they are used for a period of 6 months or until used.
- Storage Requirements: Stored in a freezer at ~ -10°C in the dark and kept for a period of one year unopened.*

Compound/Mix	Volume[1]	Concentration
Internal Standard Mix (2500 ppm)	200 uL	50 ppm
Surrogate Spiking Mix (2000 ppm)	250 uL	50 ppm

[1]Each compound/mix is diluted to 10 mLs with methanol. Total volume made may vary depending on necessity at the time it is prepared.

All standard preparation is entered into LabNet (and recorded in the Standards Preparation Logbook). All standard labels contain the following information: standard description, concentration, date prepared, analyst, and expiration date. Addition of 2.5 uL of each solution to 25 mLs of sample results in a concentration of 5 ppb per each component.

- Life of Standard: Working solutions have an expiration date of 2 weeks.
- Storage Requirements: These are stored in 1.5 - 2.0 mL amber Teflon-lined screw-capped vials at ~ –10°C in the dark.

5.4 Stock Purgeable Standards

These are obtained as neat solutions from Ultra, Supelco and Restek. The contents of each solution and concentration appear on the next page. Upon opening, all contents are transferred to 1.5 - 2.0 mL amber, Teflon-lined screw-capped vials. Listed are compounds in the EPA TCL and includes compounds analyzed on a regular basis. Other standards, if needed, are either purchased as neat solutions or neat standards from Supelco, Chem Service or other certified supplier. See appropriate entries in LabNet.

Stock Purgeables
Calibration Standards

Equity SS 8260 Calibration Mix 1 2000 ug/mL in Methanol		SS Volatile Organic Compound Mix 2000 ug/mL in Methanol
		Chloroethane
1,1,1,2-Tetrachloroethane	Dichloromethane	Methyl Bromide (Bromomethane)
1,1,1-Trichloroethane	Ethylbenzene	Methyl chloride (Chloromethane)
1,1,2,2-Tetrachloroethane	Hexachlorobutadiene	Trichlorofluoromethane
1,1,2-Trichloroethane	Isopropylbenzene	Dichlorodifluoromethane
1,1-Dichloroethane	m-Xylene	Vinyl chloride
1,1-Dichloroethylene	n-Butylbenzene	
1,1-Dichloropropylene	n-Propylbenzene	
1,2,3-Trichlorobenzene	Naphthalene	
1,2,3-Trichloropropane	o-Xylene, p-Xylene	**Vinyl Acetate**
1,2,4-Trichlorobenzene	sec-Butylbenzene	
1,2,4-Trimethylbenzene	Styrene	2000 ug/mL in Methanol
1,2-Dibromo-3-chloropropane	tert-Butylbenzene	
1,2-Dibromoethane	Tetrachloroethylene	**Volatile Ketone**
1,2-Dichlorobenzene	Toluene	Acetone
1,2-Dichloroethane	trans-1,2-Dichloroethylene	2-Hexanone
1,2-Dichloropropane	trans-1,3-Dichloropropylene	Methyl ethyl ketone
1,3,5-Trimethylbenzene	Trichloroethylene	4- Methyl-2-pentanone
1,3-Dichlorobenzene		
1,3-Dichloropropane		5000 ug/mL in Methanol
1,4-Dichlorobenzene		
2,2-Dichloropropane		**Carbon Disulfide**
2-Chlorotoluene		2000 ug/mL in Methanol
4-Chlorotoluene		
4-Isopropyltoluene		**MTBE**
Benzene		2000 ug/mL in Methanol
Bromobenzene		
Bromochloromethane		**THF**
Bromoform		2000 ug/mL in methanol
Carbon tetrachloride		
Chlorobenzene		
Chlorodibromomethane		
Chloroform		
cis-1,2-Dichloroethylene		
cis-1,3-Dichloropropylene		
Dibromomethane		
Dichlorobromomethane		

NOTE: This list contains compounds not on the TCL list for this method. However, the same standard is used for several methods. Therefore, all standards are listed here to minimize confusion. Standards are ordered from Ultra, Supelco or Restek.

5.4.1 Stock 5-Point Standard

Each transfer is entered into LabNet (and recorded in the Standard Preparation Logbook) and each standard is assigned a unique ID# (i.e., SRN). The Working Standards are prepared as follows.

Compound/TCL Mix	Volume[1] (uL)	Concentration
2000 ppm Calibration Mix 1	25	25 ppm
2000 ppm MTBE	25	25 ppm
5000 ppm KETONES	50	125 ppm
2000 ppm CS2	25	25 ppm

[1]Diluted to 2 mL with methanol.

The Stock Gases Mix is prepared as follows:

Compound/TCL Mix	Volume[1] (uL)	Concentration
2000 ppm Organic Comp. MIX	25	25 ppm

[1]Diluted to 2 mL with methanol.

The Tetrahydrofuran standard is prepared as follows:

Compound Mix	Volume[1] (uL)	Concentration
2000 ppm THF	250	500 ppm

[1]Diluted to 1 mL with methanol.

- Life of Standard: Working solutions have an expiration date of 2-weeks/1 week respectively.
- Storage Requirements: These mixtures are stored in 1.5-2.0 mL amber Teflon-lined screw-capped vials at ~ –10°C in the dark

5.5 Stock Laboratory Control Sample (LCS) / Matrix Spike (MS) Solution

Stock Purgeables LCS / Matrix Spike (MS) Standard OLC2 SPIKE 2500 ug/mL in methanol	
Carbon Tetrachloride	Tetrachloroethene
Trichloroethene	1,2-Dibromoethane
Benzene	Bromoform
1,2-Dichloropropane	1,4-Dichlorobenzene
cis-1,3-Dichloropropene	1,2-Dichloroethane
1,1,2-trichloroethylene	

Compound	Concentration
Vinyl Chloride	5000 ppm

- Life of Standard: Neat standards are kept for a period of 1-year or manufacturer's date. Once opened, the stock can be used for period of 6 months or until QC indicates a new ampule should be opened.
- Storage Requirements: These are stored at ~ -10°C in the dark prior to use.

The working MS solution is prepared as follows:

Compound	Volume (uL) [1]	Concentration
Vinyl Chloride (5000)	10	50 ppm
OLC2 Spike (2500 ppm)	20	50 ppm

[1] Each diluted to 1 mL methanol

Addition of 2.5 uLs of each solution results in all components at 5 ppb.

All standard preparation is entered into LabNet (and recorded in the Standards Preparation Logbook). See above for label information.

- Life of Standard: This solution is stored at ~ –10°C in several 1.5 -2.0 mL amber Teflon-lined screw-capped vials.
- Storage Requirements: Working matrix spike solutions have an expiration date of 1 week/2 week respectively or until low recoveries of the matrix spike compounds indicate a new solution is needed.

Only the method specified compounds are controlled on (refer to Attachment 1).

5.6 Stock BFB Solution

The BFB standard is purchased as a neat solution from Ultra.

Stock	Amount (uL)	MeOH	Concentration
BFB (2000)	25	diluted to 2 mL	25 ppm

- Life of Standard: This stock can be kept for a period of 1-year until opening. Upon opening, the solution is transferred to a 1.5 - 2.0 mL vial and assigned an SRN. Once opened, it is used for a period of 6 months.
- Storage Requirements: The standard is stored at ~ –10°C in the dark

5.6.1 Working BFB Solution

Addition of 2 uL/5 mLs results in a concentration of 50 ng/5 mLs in organic-free water (OFW).

- Life of Standard: The Working BFB Solution is used once and not stored

NOTE: Intermediate and Working Solutions are never assigned an expiration date exceeding the expiration date of the neat/stock standards/solutions. Again, if a manufacturer's date is evident, "neat" standards are assigned that date.

6.0 CALIBRATION (NON-DAILY)

Before an instrument is used as a measuring device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration is documented.

6.1 PFTBA Autotune or Manual Tune

The instrument is first tuned in one of two ways: autotune or manual tune. The ion abundances in the calibration gas are best monitored near the temperature of analysis of BFB. The ion abundances in the calibration gas are monitored at a temperature of 100°C and a column flow of 1.0 mL/minute. Monitoring at this temperature produces the most representative cal gas scan and therefore the best estimate of BFB response.

6.1.1 If an AUTOTUNE is to be performed, continue below. If not, proceed to Sec. 6.1.3. An autotune is not run before every 5-point calibration. If the instrument has been down for any reason previously listed or major difficulties in manual tune are encountered, an autotune is performed. Autotunes are generally NOT performed when an existing 5-point is being met.

6.1.2 Follow instructions and retrieve a hardcopy of the autotune results. Check the following:

- passed/fail: in itself, not necessarily an indication of MS performance
- repeller and ion focus settings
- electron multiplier voltage

Mass	Relative Abundance
69.0	100%
219.0	> 30%
502.0	> 1%

The repeller and EM voltages are good indicators of the sources' cleanliness. Generally, the lower the setting the cleaner the source. Other factors may, however, supersede (i.e., the age of the multiplier) and a clean source will not always autotune these low. The EM is set by autotune program to produce an abundance for mass 69 around 1000000. The operator can plan on having to increase this by 100-200 to achieve normal analysis sensitivity.

Observe peak shape, absence of lead-ons/tailing, the resolution between isotopes, peak width and mass axis. A hardcopy of the profile scan is desirable, and can be filed with the autotune results.

6.1.3 If an AUTOTUNE has just been performed, continue here. If not, proceed to Sec. 6.1.6. Enter MANUAL TUNE and read the autotune (which was automatically stored in a file). For volatiles, edit the scan parameters to monitor ions 69, 131 and 219.

6.1.4 Enter one of several methods available and adjust the parameters (usually the ion focus, entrance lens and amu gain) to achieve the following relative abundances:

Mass	Relative Abundance
69	100%
131	30 – 55%
219	33 – 70%

These will vary with the MS. Mass 219 is usually 5-9% greater than mass 131. Adjust the EM voltage up 100 volts. If necessary, adjust the amu gain for peak shape and high-end isotope resolution. An overall peak-width of 0.500 is desirable.

Again, these adjustments and relative abundances may not guarantee that BFB will meet requirements, but is a good place to start.

6.1.5 Hardcopy the profile scan and store the tune in the appropriate tune file. This file can serve as a diagnostic tool and can also provide a starting point in the event that the operator has trouble meeting the 5-point calibration.

NOTE: In volatiles, as opposed to semi-volatiles, there are a few more things that will affect continuing calibration. Although there may be times when adjusting the tune parameters may help, it will most likely be another problem (i.e., age of standards). Save the changes to the appropriate Tune File. Exit the program.

6.1.6 If an AUTOTUNE has not been performed, enter MANUAL TUNE and adjust any parameters, if need be. Adjustment may not be necessary, and not desirable, if problems in tuning or meeting the initial calibration have not been encountered. Hardcopy the profiles scan, store to the appropriate tune file, and exit.

NOTE: Again, this process in volatiles is not as critical as it is in semi-volatiles. It may be desirable if the operator has been having problems with the instrument, but this is not always necessary. Observing and hardcopying the profile scans does serve as a reference for the next day.

6.2 BFB Analysis

Once the instrument is tuned, a 50 ng injection of 4-Bromofluorobenzene must meet criteria. The mass spectrum must meet the following criteria:

Mass	Ion Abundance
50	8 - 40% of mass 95
75	30 - 66% of mass 95
95	Base Peak, 100% rel. abund.
96	5 - 9% of mass 95
173	<2% of mass 174
174	50 - 120% of mass 95
175	4 - 9% of mass 174
176	93.0 - 101.0% of mass 174
177	5 - 9% of mass 176

The BFB is analyzed by one of the methods in Attachment 2. **(Method parameters listed in the appendices are examples only. This statement applies to all references made to these methods).** Typical Concentrator conditions also appear in Attachment 2. The abundances of the designated masses above MUST meet the criteria before analyses can begin. If necessary, enter MANUAL TUNE, adjust parameters and reanalyze the BFB tune. The instrument is tuned every 12 hours of analysis.

The spectrum must be taken by adding three scans (Apex, just before and after) and subtracting the background (prior to elution of BFB).

6.3 Initial 5-Point Calibration

Completed:
- as needed-continuing calibration can not be met;
- after a source cleaning and/or column change; or
- any time a major repair or change has occurred with the instrument that affects calibration

When a CCV exceeds it's recommended acceptance criteria, inspect the system to determine the cause and restore original conditions. Reanalyze the standard or prepare a new standard. Document all actions / reasons in the comment section of each runlog. **Under no circumstances is a CCV to be re-run prior to completing the aforementioned reviews; or automatically running 2 CCV's.**

There are various types of instrument maintenance that should automatically require re-calibration. Examples include changing traps, bulkhead fittings, TPC valves, column fittings or filaments.

Confirm that the GC/MSD or bench-top is stable and equilibrated. If at all possible, allow the instrument to equilibrate overnight at all operating temperatures if the source/column has been cleaned/changed. Prior to beginning an initial calibration, it is a good idea to:

- Check the background of air/water levels and base ion by scanning for appropriate ions and visually inspecting the spectrum scans for any other possible or undesirable background.
- Recheck the multiplier settings, after a source is cleaned the EM can most often be dropped.

Each 5-point calibration standard is analyzed according to one of the methods in Attachment 2 – which are examples. (Refer to the STL Corporate Policy, P-T-001, *Selection of Calibration Points* for further guidance.)

Allow standards to come to ambient temperature.

Fill a 25 mL luer-lock gas-tight syringe with reagent water to overflowing. Replace the plunger and invert. Adjust to 25 mLs, confirming the absence of any air bubbles. Pull back slightly on the plunger to allow addition of standards.Use the following as a guide:

Conc. Level (Gas & OLC / Ketone / THF) (ppb)	Working 5-Pt. Stds. (Gas/OLC Std./THF) (uL)	Working ISS (uL)	Working SSS (uL)
25 / 125 / 250	25 / 25 / 12.5	2.5	12.5
10 / 50 / 100	10 / 10 / 5	2.5	5
5 / 25 / 50	5 / 5 / 2.5	2.5	2.5
2 / 10 / 20	2 / 2 / 1	2.5	10 (1/10 dil.)
1 / 5 / 10	1 / 1 / 0.5	2.5	5 (1/10 dil)

Immediately add the standards to a clean 40 mL vial. Following the parameters in Attachment 2, analyze the 5 ppm standard. A normal standard will appear very similar to the one in Figure 1. Quantitate the standard against the appropriate method file. A short list example of one file appears in Attachment 1. Sufficient areas for the first internal standard will vary somewhat between instruments. Acceptable areas should be based on maintaining sufficient sensitivity for poor responders without saturating the detector at the upper end of the calibration range.

Too low an area will almost guarantee poor/unsatisfactory responses of low-response compounds and too high an area will result in saturation of some compounds at higher levels, resulting in false low response factors at high concentrations.

Response factors are calculated by the data system as follows:

$$RF = \frac{A_x \times Q_s}{A_s \times Q_x}$$

Where:
A_x = ion abundance for analyte
A_s = ion abundance for its internal standard
Q_s = concentration of its internal standard
Q_x = concentration of analyte

(Response Factors have no units)

The appropriate quant ion must be in the method. See an example of a full-listed file in Attachment 1. A listing of the target compounds with their appropriate internal standards also appears in Attachment 1. Confirm the presence of all targets and the separation of non-co-eluting compounds.

Note the response factors for the gasses. If necessary, prepare new standards.

If adjustments to the acquisition parameters are necessary, make them and re-analyze the 5 ppm standard.

After a good standard is analyzed, it is desirable to update the RF's in the ID file. This 1) will tell the operator at a glance if one or more standards is low/high and 2) could indicate that one or two were made incorrectly (not necessarily the same thing.) When a standard is analyzed and processed on target as part of the initial calibration, the RF's are automatically updated in the daily method. After all initial calibration standards are processed, checked and confirmed as being accurate and passing method criteria, the initial calibration is saved to the source method. This ensures that the correct initial calibration is used for each ensuing continuing calibration check. A hardcopy of the calibration report is generated. All minimum RF's and maximum %RSD for the required compounds is confirmed. (Limits listed in Attachment 1.) Two compounds may be out but must be <40% RSD. An example of an acceptable initial calibration appears in

Attachment 1. The BFB tune, the calibration report, all standard raw data and before and after manual integrations are kept in a file. Each instrument has its own initial calibration.

7.0 PROCEDURES

7.1 Quality Control Checks

QC Standard	Frequency	Control Limits[1]
Method Blank (MB)	Prior to analysis	All target compounds must be < CRQL except for methylene chloride, cyclohexane, acetone and 2-butanone which must be < 3X its CRQL.
Lab Control Sample (LCS)	Immediately following the MB; 1 per batch.	All recoveries must meet limits in Attachment B for the analysis to proceed.
Matrix Spike (MS) / MS Duplicate (MSD)	1 per batch ($\leq$ 20 samples)	All recoveries must meet limits in Attachment B.
Surrogates	Every MB, Sample and QC Sample	All recoveries must meet limits in Attachment B.

[1] Refer to Section 8 for complete details on control limits and corrective action procedures.

Once the MB analysis is complete and acceptable, an LCS is analyzed. Although not required by the method, an LCS is analyzed immediately following the MB.

7.2 Sample Preservation and Storage

Sample containers, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance and/or specific contract or client requests. Listed below are the holding times and the references that include container and preservation requirements for compliance with the U.S. EPA CLP Document No. OLC02.1.

Matrix	CLP	Preservation
All	10 days (VTSR)[1]	HCl, pH < 2; Cool, 4 $\pm$ 2°C

[1] VTSR = verified time of sampling receipt.
14 days holding time is used for non-CLP deliverables.

7.3 Sample Preparation

Not Applicable.

7.4 Daily or Continuing Calibration

Continuing calibration occurs prior to analysis.

NOTE: If time remains after the initial calibration, and the 5 ppb standard meets continuing calibration criteria, samples can be analyzed up to the 12 hour tune limit. The samples are quantitated against the continuing calibration standard, unless client specific method requires differently.

After having satisfied BFB tune requirements, a continuing calibration standard must be analyzed. Analyze a 5 ppb standard (25 ppb for the ketones, 50 ppb for THF) following the procedure outlined above. Confirm that all requirements in Attachment 1 are met. If so, the continuing calibration is acceptable and analysis can begin.

As with the initial calibration, the RF's are automatically updated in the daily method. If continuing calibration can not be met, either new standards and/or a new 5-point calibration is needed.

All internal standard areas and retention times are assessed immediately after calibration. Areas and times are recorded in the appropriate instrument logbook and a comparison is made to the previous days calibration. Internal standard areas should not deviate by ±40% or the retention times must be within ±0.33 minutes. If they do, appropriate action and documentation are completed.

7.5 Preventive Maintenance

Required maintenance may be performed for a variety of reasons. Certain trouble-flags will indicate what maintenance procedures may be required. A description of the situation, actions taken and follow-up must be documented in the instrument maintenance logbook on the day of maintenance and initialed / dated. An entry number is assigned to each maintenance procedure performed and this number is transferred to the appropriate instrument logbook for traceability purposes.

7.5.1 Daily Maintenance

The most routinely performed maintenance includes:

- Purge-line or sample transfer line rinses within the concentrator and Archons
- Analysis of blanks after high level samples
- GC oven bake after high level samples

7.5.2 "As Required"

Most maintenance is performed on an "as needed" basis, is operator determined and can be categorized as GC, Concentrator, MS or Archon related.

7.5.2.1 GC Related

- change column; condition new column;
- clean separator; change separator;
- check helium flow rate; and
- change gas cylinders and moisture trap.

7.5.2.2 MS Related

- clean source/rods and anything associated with that activity.

7.5.2.3 Concentrator Related

- change transfer line; clean transfer line;
- replace trap; condition new trap;
- refurbish Concentrator;
- check purge pressure and flow rate;
- analyze position blanks after high level samples; and
- change bulk head fitting.

7.5.2.4 Archon Related

- change sparge needle
- change pencil filters
- flush standard pickups
- calibrate standard valve
- run vial position calibration
- clean transfer rods
- oil bearings

7.6 Sample Preparation / Screening /Analysis

All samples are screened on a 5890 GC interfaced with a 5972 MS prior to analysis. In some cases, the GC/FID analysis can be used as a screen. Once the samples are logged into the database upon receipt, a paperwork trail is initiated. The Section Manager prints and prepares the necessary information (Sample Tracking Sheets/Big Boards) and places it in the appropriate file bin in the GC/MS VOA lab. The analysts take this information and subsequently screen the associated samples. The actual screening procedures vary due to sample appearance, sample matrix, client history and analytical method. Once the samples are screened, the paperwork is transferred to a second file appropriately labeled. This file contains information about samples that have been screened but need to be reviewed. Once screened, an 'X' is placed on top of the vial, indicating both that the sample has been screened, and that the particular vial can not be used for subsequent analysis. The screened analysis can be reviewed on screen or hard-copy, as all screening data is collected and stored on the data system as with all GC/MS analyses. Upon review, the analyst makes decisions concerning the screen and indicates if an initial dilution is required. This information is physically recorded on the paperwork. Once reviewed, the paperwork is then placed in appropriate files that are broken down by matrix and method. The samples are now ready to be analyzed.

Prior to analysis for that day, the trap should be pre-conditioned by back-flushing with Helium for 10 minutes at 180 °C.

As stated in Section 7.6.1, the MB must first be analyzed and shown to be free of interferences and target compounds. Once completed and acceptable, the LCS is analyzed. Once acceptable, the sequence of samples can be analyzed with a MS/MSD per batch of 20 or fewer samples.

7.6.1 Method Blank (MB)

Prior to any analysis, the reagent water must be shown to be free of interferences and target compounds.

To a 25 mL portion of reagent water, 2.5 uL of ISS/SSS solution are added. It is then analyzed using one of the methods in Attachment 2. All target compounds must be less than the quantitation limit. In extenuating circumstances, analysis may continue. Qualification must be made as to the positive hits above the CRQL for the compounds in question. Any associated samples containing the same compound in question must be listed in the logbook and the situation addressed in the case narrative. Once the MB analysis is complete and acceptable, analysis can proceed.

7.6.2 Laboratory Control Sample (LCS)

Immediately following the MB, the LCS is analyzed. To a 25-mL portion of reagent water, 2.5 uL of ISS/SSS and 2.5 uL of each spiking solution are added. It is analyzed and the compounds listed in Attachment 1 are controlled upon using the limits listed.

7.6.3 Sample Analysis

7.6.3.1 Allow samples and standards to come to ambient temperature.

7.6.3.2 Remove the plunger from a 25 mL luer-lock gas-tight syringe and fill to near over-flowing. Replace the plunger. Touch the tip of the syringe to a small piece of pH paper. Record the pH (Y if </=2, enter estimated pH if >2) in the appropriate column in the appropriate logbook or on the tune form. Invert the syringe, and adjust the volume to 25 mL. Confirm the absence of all air bubbles. Draw back slightly on the plunger. Add 2.5 mL of the working ISS/SSS solutions. Immediately add the sample to a clean 40mL vial. Using the methods described in Attachment 2 analyze the sample.

7.6.3.3 Any sample that contains targets above the calibration range is diluted to accurately quantitate those compounds. Any sample that has shown from past experience to be high in background/targets, or appears to be so, is screened and analyzed at a dilution.

7.6.3.4 If dilutions are to be made, the appropriate sample volume is added directly to an appropriate amount of reagent water in the 25 mL syringe. If the required amount of sample is less than 1 mL, an initial dilution is made first in a volumetric flask, and a portion of this is added to the reagent water/ISS/SSS mix in the 25 mL syringe.

7.6.3.5 Using those parameters listed in Attachment 2, analyze all samples.

7.6.3.6 Opened sample vials are used only once unless any necessary dilutions/reruns are performed the same day or there are no other vials for that sample.

7.6.3.7 If a batch of samples is to be analyzed, prepare each as above. After the batch is loaded, replace all samples and standards in storage.

7.6.3.8 Any sample that contains targets above the calibration range is diluted to accurately quantitate those compounds.

7.7 Documentation/Tracking of Sample Analyses

1. The preparation and analysis is recorded in the GC/MS Volatiles Logbook (Attachment 3), and must be completed for each day's analysis.

2. The GC/MS VOA lab employs several forms that serve both a tracking and review function. The Sample Tracking Sheet (BigBoard) is filled out for each job. It contains information the analyst needs as for method, QC requirements, special reporting requirements, screening results etc.., in addition for space to track the analysis of every single sample in the job and the outcome of that analysis.

3. The Tune Form is filled out for every 12-hour tune and contains several kinds of information. The forms main function is to track the analysis of all the samples analyzed during the 12-hours, initial review and data crunching documentation for the samples in the batch, tune and standard information etc. The Tune Form is not necessarily specific to a single job. The Tune Form is discussed again in the initial review section.

4. In addition, all samples logged into the department appear on a hold-time summary sheet where ALL samples in-house are listed by hold-times and due dates. This summary is utilized by the analysts when making decisions as to methods and analyses that are needed for the day. As samples are analyzed and reviewed, the summary sheet is constantly updated to reflect those samples completely analyzed, those requiring dilutions and re-analyses (essentially a posted summary of the Sample Tracking Sheets). At the beginning of each day, the completed analyses are removed from the summary

sheet by updating the analyzed samples into LabNet. The Sample Tracking Sheet and Tune Form can be found in Attachment 3.

7.7.1 Traceability of Standards

Upon receipt, each chemical is recorded in LabNet and is issued a unique ID#. The manufacturer, lot #, date received, expiration date, and the initials of the recording analyst are documented in LabNet. When a standard is prepared at the laboratory, the Source ID# and weight of the chemical, the type and volume of solvent, concentration, date of preparation, date of expiration, preservative if applicable, and the analyst's initials are recorded in LabNet. Each standard is given a unique ID#.

7.7.2 Data Review

Analytical data goes through a 200% review cycle. The analyst and a trained data reviewer perform the reviews according to the criteria established on the data review form (Refer to Attachment 4). Upon the first 100% review, the review form is initialed and dated as reviewed. The package, with its review sheet, comments and any CARs are submitted to the section manager or peer reviewer for a second review. Once again, the review form is initialed and dated by the second reviewer. The completed data review form remains on file with the original data.

8.0 QUALITY CONTROL

8.1 QC Summary

Quality Control is accomplished through 1) daily tuning and calibration checks and 2) preparation QC traceable through individual batches.

8.1.1 Initial Calibration

PFTBA BFB TUNE	Prior to 5-Point Curve	*Limits listed in Section 6.
25/125/250 ng \\ 10/50/100 ng | 5/25/50 ng | 2/10/20 ng | 1/5/10 ng /	5-Point Need dependent on situation.	*Refer to Sections 6.

8.1.2 Daily Analysis

PFTBA BFB	Prior to continuing calibration	*See above
Daily Calibration Standard	Prior to samples	*See Section 6.
Samples *		

*Any given 12 hour period contains a tune, standard, blank and LCS. Preparation QC is at a 5% frequency. Instrumental controls are outlined above and further discussed in the procedure section. If ever the situation arises where there is insufficient sample volume to analyze an MS/MSD at a 5% frequency then, an LCD is run, documentation of the situation is recorded in the logbook, and an SDR is completed.

Prep QC	Frequency
MB	Prior to analysis
LCS	1 per analysis batch
MS/MSD's	at least 1 set in 20
Surrogates	every blank, sample and QC Sample

The sample selection for MS/MSD are rotated among client samples so that various matrix problems may be noted and/or addressed.

The department will review the quality controls as follows:

8.1.3 At least one MB and one LCS will be included in each laboratory batch. Regardless of the matrix being processed, the LCS and MB will be in an aqueous media.

8.1.4 The MB will be examined to determine if contamination is being introduced in the laboratory.

8.1.5 The LCS will be examined to determine accuracy.

8.1.6 Accuracy will be measured by the percent recovery (%R) of the LCS. Limits are listed in Attachment 1. As stated, those compounds not specified as spikes in the method are not controlled.

8.1.7 Precision will be measured by the reproducibility of the LCS samples and will be calculated as Relative Percent Difference (RPD). Limits are listed in Attachment 1 for the LCS. RPD's are not used for bench level control or corrective action. As stated, RPD data may be used to monitor precision and generate in-house limits. At QA/QC's discretion current RPD's will be re-generated. At that time, any needed sets of LCS and LCD's will be logged into the system and analyzed. Any other time, only one LCS is analyzed with a batch.

8.1.8 Surrogate compounds will be added to every sample to measure performance of the analysis. Results must agree within statistical control limits in order to be considered acceptable. Surrogate limits are listed in Attachment 1.

8.2 Corrective Actions

Listed below are the steps that MUST be taken when an out-of-control situation occurs:

- demonstrate that all of the problems creating the out-of-control situation were addressed;
- document the problem and the action that was taken to correct the problem;
- document that an in-control situation has been achieved; and
- receive approval (signature) of the supervisor, section manager, project manger, QC personnel or other qualified personnel prior to release of data associated with the problem.

As noted, corrective action logbooks are located within each analysis log. In addition, the sample tracking form (big board), specific to a unique Job # 's is used as a CAR Form for the samples in that Job. The logbook and sample tracking form are used to note all out-of-control events, the actions taken to try and correct the problem, the return to control and qualification of data if needed.

Discussed below are the suggested and required courses of action when an out-of-control situation has occurred.

8.2.1 BFB Criteria

If BFB criteria can not be met, determine if the source of the problem is instrumental or tune related. Inspect overall sensitivity, possible excessive background, the proportionality of the masses, relative abundances of the target masses. If it seems tune related, adjust the tune parameters in Manual Tune slightly, until acceptance is achieved. If the problem seems instrumental, perform suggested trouble-shooting to locate and correct the problem (Suggestions can be found in most of the manuals). NO analysis can proceed until criteria are met. Each instrument will have its own idiosyncrasies.

8.2.2 Initial Calibration

If initial calibration can not be met, determine if the problem is analytical or instrumental. Some suggested questions to ask would be:

- were the standards prepared correctly?
- was the proper amount analyzed?
- check the chromatogram - did something happen on one or two analyses; i.e., a leak
- check the response factors; is one concentration level very high or low? reanalyze it how old are the standards?

The course of action depends on the problem. Criteria are listed in Attachment 1.

8.2.3 Continuing Calibration

If continuing calibration can not be met, determine if the problem is analytical or instrumental. Some suggestions:

- check the chromatography
- is overall sensitivity low?
- excessive background?
- how old is the standard?
- need a new 5-point?
- has the tune shifted?

Compare the relative abundances of 69, 131 and 219 from that day's manual tune to those on the day the initial calibration was analyzed. Slight adjustments to the tune may bring the standard in. If adjustments are made to the tune a new BFB tune must be analyzed and pass before running another continuing calibration.

Certain compounds will help indicate what the problem is. The course of action depends on the problem. Criteria are listed in Attachment 1.

8.2.4 Surrogates

If ANY surrogates are outside limits in the MB, it must be re-analyzed. Analyses CAN NOT proceed until an in-control situation is demonstrated. Re-analyze the blank. If surrogates are still out, the instrument may need to be re-tuned (BFB) and/or another calibration standard analyzed. If the problem persists, further maintenance action may be required (i.e., trap replacement, clean separator).

Before pursuing other measures, check to be sure that:

- calculations are correct
- concentrations of the surrogates in the spiking solution are correct
- the correct amount of ISS/SSS solution was added
- ISS/SSS areas are reasonable

If any surrogates in a sample are outside limits, check the above first. Any sample that has a surrogate out must be re-analyzed. The re-analysis can take the form of a dilution. If the surrogate (s) is/are still outside limits a matrix effect is demonstrated and both reports are submitted.

If all surrogates are in-control on the re-analysis, only the second analysis is reported.

Every effort is made to complete the re-analysis within hold-time. If this is impossible (i.e., capacity hold-times preclude re-analyses hold-time) both reports may be submitted. This is documented in the narrative.

If the sample with the out-of-control surrogates is the same sample on which the MS and MSD has been performed, and the pattern is duplicated, then re-analysis is NOT required. Documentation of the similarities is required. MS and MSD recoveries and RPD's are advisory only. However, if problems are frequent, investigation of the system is necessary.

8.2.5 Method Blank (MB)

If the MB is contaminated, re-analyze it on a different position.

If contamination is still present, the problem may be in one of the common elements, such as the trap, transfer line, port valve or column. Baking the trap/column and running position blanks may be necessary. If contamination has occurred beyond that, and maintenance is required (i.e., replace trap) it is documented in the Maintenance Logbook. Analysis CAN NOT proceed until the blank is free of contamination. In extenuating circumstances, analysis may continue. Qualification must be made as to the positive hits above the CRQL for the compounds in question. Any associated samples containing the same compounds in question must be listed in the logbook and the situation addressed in the case narrative.

8.2.6 LCS's

Current limits are those listed in Attachment 1 for the 12 method-specified MS compounds. Analysis can not proceed until the LCS meets limits. Possible courses of action may include re-calibrating, preparing either new calibration standards or MS standards. The course of action will depend on the problem. Outliers are documented and corrective action evaluated at that time.

8.2.7 Matrix Spikes (MS)

MSs are not required by this method but can be performed upon client request. The following applies to MSs:

MS and MSD recoveries and RPD's are advisory only and function to illustrate matrix effects on the efficiency and completeness of target analyses. Outliers are documented. Current limits are listed in Attachment 1.

As stated, all method criteria as described in this SOP are evaluated prior to analysis. Out-of-control events are documented immediately and action taken at the time to return to control. In extenuating circumstances (i.e., hold-time issues), sample analysis may proceed. The logbook contains a CAR section to document this and an additional section that qualifies any affected data. Any affected data is documented in the case narrative. Certain events may not affect usability of the data.

A general maintenance logbook does exist for each instrument. An entry number is given to each maintenance action performed. This number is then transferred to the appropriate instrument logbook. Major maintenance and problems are noted in this logbook. Other courses of action taken for out-of-control situations are documented either on the review form or the case narrative.

8.2.8 Internal Standards

OLC02.1 requires that all internal standards meet criteria. Re-analysis is required for any sample not meeting the requirements. This is documented on the sample CAR form. If the sample with out-of-control areas is the same sample the MS and MSD were performed on, and the pattern is replicated, re-analysis is not required. The replication is noted and narrated.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Computer Data Production/Reduction

The Target software produces a Total Ion Chromatogram (TIC), header, quant report and background subtracted spectra. For those clients requiring it, a 30 tentatively identified compound (TIC) search is also performed. The data system will produce an integration listing and tentative identification of each hit found at the selected percentage of the largest peak present. All data for each sample is kept together. All data for all samples run during that 12-hour period remains together until operator data work up begins. At which time all appropriate QC is photocopied for each Job # in that run batch.

9.2 Quantitation of Target Compounds

Quantitation of the target compounds is performed by the data system but can be accomplished as follows:

$$\text{Concentration (mg/L)} = \frac{[A_x \times I_s]}{[A_{is} \times RF]} \times DF$$

Where:
A_x = area of characteristic ion for target
I_s = concentration of internal standard (ng)
A_{is} = area of characteristic ion for int. std.
RF = response factor for target
DF = dilution factor (if any)

The sample volume is considered to be "constant" for calculation purposes, and is always entered as 25 in the LabNet. Less sample volume is taken into account in the dilution factor.

9.3 Quantitation of TIC's (Tentatively Identified Compounds)

The Target processing software performs quantitation of TIC's. The formulas above for waters can be used with the following modifications. A_x and A_{is} should be taken from the total ion integration listing accompanying the TIC report produced by the data system. The nearest non-interfered with internal standard should be used. The RF is assumed to be one (1). The concentration is therefore an estimate and is flagged as such with a "J". Any TIC also found in the MB is flagged with a "JB". Any TIC identified with a CAS number is also flagged with a "N" to indicate identification is based on the mass spectra. The operator should visually confirm that the integration is correct. If not, the peak in question must be manually integrated. The target system automatically calculates the actual concentration of the TIC's, including dilutions and total solids, once that information is retrieved from LabNet.

9.4 Operator Data Reduction/Review

The operator does on-screen review of all data and 1) makes judgments concerning the "realness" of those target compounds found and 2) makes judgments concerning the identification of the tentatively identified compounds and 3) modifies the output to produce a data package reflective of those decisions.

9.4.1 Initial Review

Operator determines that the analysis in itself is acceptable. This means QC Samples/Criteria are met:

- The MB contains no interferences or target compounds at the CRQL;
- ALL surrogates are in control in the blank (Limits are listed in Attachment 1);
- ALL surrogates in samples are in control (Limits are listed in Attachment 1);
- LCS recovery limits are met. (Limits are listed in Attachment 1); and
- Internal standard areas and retention times are checked and meet limits. (Limits are listed in Attachment 1)

NOTE: The LCS is analyzed immediately after the blank. All recoveries must meet limits for analysis to proceed. There are exceptions to this procedure (hold-time situations) and these would be documented on the CAR form. CAR forms are found within each logbook. Outliers for surrogates, LCS's and internal standards are immediately noted on a CAR form. Corrective action is decided upon at that time. An example of CAR forms appears in Attachment 3.

- The sample does not require any further dilutions or analysis at a more concentrated level. Visually confirm complete integration for any large and/or saturated target compounds. Dilutions are made to keep the target in the upper half of the calibration range. The MS and MSD are never diluted to get spiked or non-spiked compounds within range, as this would reduce the matrix affect assessment.

- The sample does not require re-analysis for any other reason (i.e., leak, analysis past tune time, ISTD areas low, etc...).

9.4.2 Identification of Targets

The following guidelines are used in the positive identification of target compounds.

"elution of component at the same relative retention time as the standard component."

The elution times should compare within ±30 s. The standard must be run on the same 12 hour period as the sample. If co-eluting analytes interfere with the comparisons of retention times, other ions characteristic to that compound can be used to confirm relative retention times.

"correspondence of the sample component and standard component mass spectrum." Comparisons of sample spectra to standard spectra must be made using standard spectra obtained from the GC/MS system.

All ions present in the standard spectrum at a 10% relative intensity (most abundant ion being 100%) MUST be present in the sample.

The relative intensities of the above ions must agree within ± 20%, between the standard and sample. If an ion is 50% intensity in the standard the corresponding ion must be between 30 and 70% in the sample.

Ions >10% in the sample but not present in the standard must be considered and accounted for.

Operator judgment. If a compound can not be verified by the above, but in the operators technical judgment the ID is correct, it is reported as such.

Once all positive identification is made, the file is modified to reflect these decisions. At this time, TIC's may also be reviewed and named. In each case where the file has been edited or manual integrations have taken place, the operator must identify, initial and date the changes on the hardcopy. The following guidelines apply and are additional discussed in the Manual Integration Policy SOP (S-Q-004):

- Manual integrations should be consistent between all files integrated.
- Manual integrations should not be performed to meet QC criteria.
- Manual integrations are automatically flagged with an "M" on the raw data.
- Manual integrations should be labeled with a reason for the integration.
- Excessive manual integrations may reflect an instrumental or methodological problem that should be addressed.

Manual integrations are most often performed for the following reasons.

- Assignment of correct peak that was mis-identified by the system.
- Incomplete auto-integration due to high level of target detected.
- Incomplete auto-integration due to background interference.
- Incorrect auto-integration due to co-elution or near co-elution of compounds.
- Missed peaks.

All manual integrations are reviewed, initialed and dated. For those clients requiring full data packages, spectra and Extracted Ion Chromatography Profiles (EICP) are printed for all manually integrated compounds. Manual integrations are documented in the case narrative and a Manual Integration Summary is included in the data package.

9.4.3 Identification of TIC's

In general, up to as many as 30 non-target compounds are tentatively identified by the data system. Compounds with responses <40% of the nearest ISTD are not identified. The data system provides the operator with a SUB ADC C sample spectrum, spectra of the first three matches and a listing of two other possibilities. Molecular formulas, molecular weights and CAS #'s are included. The following guidelines are used:

Relative intensities of major ions in the reference spectrum should be present in the sample (ions >10%).

- Relative ions should agree within $\pm$ 20%.
- Molecular ions in the reference should be in the samples.
- Review the possibility of background and/or co-eluting compounds for those ions present in the sample but not in the standard.
- If ions are present in the sample but not in the standard, review the possibility of the presence of background or co-eluting compounds.
- If ions are present in the standard but not in the sample, review the possibility that the ions were subtracted out because they are also common to the background or co-eluting compounds.
- In the event no valid interpretation can be made, the compound is called "unknown".
- Interpretation can be often narrowed down to a class of compounds, molecular formula or weight.

9.5 Final Review/Package Preparation

Once the analysis is determined to be acceptable, the initial review and data reduction has occurred and the analyst has entered sample prep info into Labnet, then, the analyst prints hard copies of all the necessary raw data. The analysts review the hard copies and initial and date them, documenting that review. All required forms are then generated using the Target software. The package is then assembled and ready for the first review.

Upon the first 100% review, the review form is initialed and dated as reviewed. The analyst preparing the data package normally does the initial review. The package, with its review sheet, comments and any CAR forms is submitted to the supervisor, section manager, or secondary analyst for a second review and validation. Once the data passes review in the department, it is submitted to report generation/QA/QC for appropriate follow-up action. The complete analysis scheme and sample tracking system are summarized in Attachment 5.

9.6 Data Management

9.6.1 Archival of Data

There are three full back-ups performed per week.

- Every Thursday a full back up of VOA data is performed.
- Every Friday a full back up of SVOA data is performed.
- Every Tuesday a system back up (minus the NBS Library) is performed. There are two tapes provided for this back up, and are rotated each week. Most current tapes are kept off site. Older tapes are in locked storage.

The system maintains a database, or logs, of each back-up session. Successful completion of each back-up can be verified each morning by accessing the job report logs in ARCServe. This is performed each morning. Any missed jobs can be rescheduled and completed in the morning of the following day. As noted above, this database is re-archived after every normal back-up and can be retrieved at any time necessary.

9.6.2 Removal of Data

Although there is a substantial amount of space available to both BNA's and VOC's during busy periods the system can fill rather quickly. As an estimate, with a total of (17) instruments, a maximum of about 2-3 months (per instrument) can be kept on the system at one time. There is not necessarily a set definite schedule of removing data from the system. Once the data package has been completed and all data associated from that batch has been reduced, reviewed, packaged and sent to report generation, the tune form is placed in designated location. Either by necessity or at the supervisors discretion, these are compiled and the data then actually removed from the system.

The tune forms are then filed in the office area. Once a year, these forms are compiled and boxed and stored in a general data storage area.

10.0 WASTE MANAGEMENT AND POLLUTION CONTROL

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Laboratory generated aqueous waste will enter the "Waste Water" waste stream
- Expired Standards waste from this procedure will enter the "Flammable Vials" waste stream.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Figure 1. Example: 5 ppb Standard on Capillary Column

Table 1. Contract Required Quantitation Limits (CRQL)
Table 2. Retention Times and Characteristic Ions for Volatile Compounds

Attachment 1. Examples: ID File Listing; Target Compounds and Internal Standards; Initial/Continuing Calibration Requirements; Initial Calibration/ Continuing Calibration; Surrogate Recovery Limits; LCS / MS Recovery Limits
Attachment 2. Example: Method Listings; Concentrator Conditions; Flow Settings
Attachment 3 Examples: Analysis / CAR-Qualification Logbook; Maintenance Logbook; Tune Form; Sample Tracking Sheet; Corrective Action Report Form
Attachment 4. Example: Data Review Checklist
Attachment 5. Analysis and Sample Tracking Flowcharts

Historical File:

Revision 00: 11/27/96
Revision 01: 06/25/99
Revision 02: 09/22/00
Revision 03: 08/18/03
Revision 04: 07/16/04

Reason for Change; Revision 03:

- General procedure review and update.
- Updated Sections 3 & 10 on Safety & Waste Disposal.
- General procedure review and update for all sections.

U:\QC\SOP\MV\OLC21.DOC

Figure 1.

Example: 5 ppb Standard on Capillary Column

INITIAL CALIBRATION DATA

Start Cal Date : 04-FEB-2004 01:38
End Cal Date : 04-FEB-2004 03:21
Quant Method : ISTD
Origin : Disabled
Target Version : 3.50
Integrator : HP RTE
Method file : /var/chem/gc13.i/020404_olcprc3w.b/olcprc3w.m
Cal Date : 20-Jul-2004 14:07 petruszj
Curve Type : Average

Calibration File Names:
Level 1: /var/chem/gc13.i/020404_olcprc3w.b/3i0204.d
Level 2: /var/chem/gc13.i/020404_olcprc3w.b/3i0204a.d
Level 3: /var/chem/gc13.i/020404_olcprc3w.b/3i0204b.d
Level 4: /var/chem/gc13.i/020404_olcprc3w.b/3i0204c.d
Level 5: /var/chem/gc13.i/020404_olcprc3w.b/3i0204d.d

Compound	1.000 Level 1	2.000 Level 2	5.000 Level 3	10.000 Level 4	25.000 Level 5	RRF	% RSD	
1 Dichlorodifluoromethane	0.39095	0.40097	0.44606	0.51299	0.47593	0.44538	11.471	
2 Chloromethane	0.24451	0.24872	0.25936	0.27563	0.26088	0.25782	4.707	
3 Vinyl chloride	0.30765	0.31189	0.30804	0.33033	0.31877	0.31534	3.012	
4 Bromomethane	0.18350	0.18253	0.20634	0.22031	0.21478	0.20149	8.729	
5 Acetonitrile	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
6 Vinyl Acetate	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
7 Acrolein	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
8 Chloroethane	0.18688	0.18541	0.18737	0.19556	0.18380	0.18780	2.425	
9 Trichlorofluoromethane	0.58227	0.61251	0.56024	0.62728	0.59050	0.59456	4.401	
10 1,1-Dichloroethene	0.28661	0.30234	0.27667	0.30222	0.28344	0.29025	3.980	
11 Acetone	0.01473	0.01056	0.00955	0.00978	0.00895	0.01071	21.623	<-ok
12 Carbon Disulfide	0.51910	0.52333	0.48839	0.52173	0.48987	0.50849	3.488	
13 Acrylonitrile	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
14 Methylene chloride	0.29612	0.26769	0.23659	0.24168	0.22292	0.25300	11.486	
15 trans-1,2-Dichloroethene	0.36288	0.34755	0.33111	0.34931	0.33491	0.34515	3.663	
16 Methyl-tert-Butyl Ether	0.25634	0.24944	0.24257	0.25173	0.23558	0.24713	3.297	
17 Trichlorotrifluoroethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
18 1,1-Dichloroethane	0.61989	0.62617	0.60507	0.63212	0.60555	0.61776	1.969	
19 cis-1,2-Dichloroethene	0.30521	0.30328	0.29867	0.30629	0.29303	0.30130	1.814	
20 2,2-Dichloropropane	0.48966	0.50712	0.46439	0.49998	0.47196	0.48662	3.729	
21 2-Butanone	0.01305	0.01389	0.01720	0.02005	0.02000	0.01684	19.596	
22 Bromochloromethane	0.14822	0.14437	0.14314	0.14993	0.13984	0.14510	2.780	
23 Tetrahydrofuran	0.01311	0.01324	0.01309	0.01414	0.01416	0.01355	4.087	
24 Chloroform	0.53364	0.55173	0.53481	0.56414	0.53512	0.54389	2.494	
25 2-Propanol	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
26 1,1,1-Trichloroethane	0.72195	0.82882	0.73933	0.79317	0.78085	0.77283	5.537	

INITIAL CALIBRATION DATA

Start Cal Date : 04-FEB-2004 01:38
End Cal Date : 04-FEB-2004 03:21
Quant Method : ISTD
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Target Version : 3.50
Integrator : HP RTE
Method file : /var/chem/gcl3.i/020404 olcprc3w.b/olcprc3w.m
Cal Date : 20-Jul-2004 14:07 petruszj
Curve Type : Average

Compound	1.000 Level 1	2.000 Level 2	5.000 Level 3	10.000 Level 4	25.000 Level 5	RRF	% RSD	
27 1.1-Dichloropropene	0.52833	0.55286	0.48807	0.54469	0.51506	0.52580	4.881	
28 Carbon tetrachloride	0.78524	0.87338	0.76732	0.85290	0.82669	0.82111	5.431	
30 Benzene	1.13961	1.27672	1.17492	1.22615	1.22347	1.20817	4.353	
31 1.2-Dichloroethane	0.17687	0.18459	0.17702	0.18359	0.17323	0.17906	2.708	
32 Crotononitrile	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
34 Propionitrile	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
35 Ethyl ether	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
36 Ethyl Acetate	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
37 2-Chloro-1.3-butadiene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
38 Trichloroethene	0.59944	0.68999	0.63162	0.66920	0.66593	0.65124	5.487	
39 2-Nitropropane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
40 1.2-Dichloropropane	0.41797	0.46952	0.43316	0.44999	0.44070	0.44227	4.346	
41 Dibromomethane	0.19514	0.21182	0.20744	0.21492	0.21218	0.20830	3.759	
42 Bromodichloromethane	0.54215	0.62884	0.59397	0.61674	0.61227	0.59880	5.687	
M 43 1.2-Dichloroethene (total)	0.33405	0.32542	0.31489	0.32780	0.31397	0.32322	2.670	
44 Hexane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
45 3-Chloropropene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
46 cis-1.3-Dichloropropene	0.43534	0.50475	0.48474	0.51297	0.51817	0.49120	6.864	
47 4-Methyl-2-pentanone	0.07122	0.08548	0.08640	0.08979	0.09123	0.08482	9.387	
49 Toluene	0.73832	0.84441	0.78044	0.82208	0.81682	0.80041	5.200	
50 Iodomethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
51 Bis(chloromethyl)ether	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
52 trans-1.3-Dichloropropene	0.27751	0.31302	0.30026	0.32837	0.32515	0.30886	6.714	
53 tert-Butyl alcohol	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
54 1.1.2-Trichloroethane	0.18537	0.21304	0.19303	0.19478	0.19406	0.19606	5.211	
55 Tetrachloroethene	0.63602	0.74318	0.65383	0.71565	0.70483	0.69070	6.445	
56 1.3-Dichloropropane	0.28887	0.32863	0.31394	0.32660	0.32130	0.31587	5.105	
57 Isobutanol	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
58 2-Hexanone	0.02567	0.03875	0.04445	0.05160	0.05309	0.04271	26.059	
59 Methacrylonitrile	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
60 n-Butanol	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
61 2-Chloroethylvinylether	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
62 Dibromochloromethane	0.40859	0.46012	0.42846	0.46953	0.45760	0.44486	5.718	

INITIAL CALIBRATION DATA

Start Cal Date : 04-FEB-2004 01:38
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Quant Method : ISTD
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Target Version : 3.50
Integrator : HP RTE
Method file : /var/chem/gc13.i/020404 olcprc3w.b/olcprc3w.m
Cal Date : 20-Jul-2004 14:07 petruszj
Curve Type : Average

Compound	1.000 Level 1	2.000 Level 2	5.000 Level 3	10.000 Level 4	25.000 Level 5	RRF	% RSD	
63 1,2-Dibromoethane	0.24334	0.28390	0.27368	0.29176	0.28582	0.27570	6.975	
64 1-Chlorohexane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
66 Chlorobenzene	0.91862	1.02354	0.95680	1.01301	1.00164	0.98272	4.471	
67 1,1,1,2-Tetrachloroethane	0.43882	0.49748	0.47142	0.49365	0.48653	0.47758	4.994	
68 Ethylbenzene	0.44289	0.49998	0.46728	0.50527	0.49787	0.48266	5.541	
69 p,m-Xylene	1.15233	1.27772	1.18912	1.29016	1.26557	1.23498	4.916	
70 o-Xylene	1.04726	1.19673	1.09801	1.18848	1.17145	1.14039	5.703	
71 Styrene	0.69747	0.81743	0.79167	0.84034	0.84950	0.79928	7.652	
72 Methylmethacrylate	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
73 Bromoform	0.39731	0.40518	0.41941	0.44880	0.43106	0.42035	4.883	
74 Isopropylbenzene	3.44998	3.47748	3.26012	3.50756	3.40345	3.41972	2.839	
76 1,1,2,2-Tetrachloroethane	0.20399	0.23777	0.21956	0.23949	0.23056	0.22627	6.506	
77 Bromobenzene	0.80371	0.83944	0.83013	0.87766	0.84801	0.83979	3.205	
78 1,2,3-Trichloropropane	0.10622	0.10663	0.10262	0.10677	0.10291	0.10503	1.982	
79 n-Propylbenzene	3.75994	3.86852	3.78273	4.10707	4.09393	3.92244	4.272	
80 2-Chlorotoluene	2.75150	2.66055	2.59033	2.76212	2.83675	2.72025	3.523	
81 1,3,5-Trimethylbenzene	2.72318	2.66307	2.53459	2.76389	2.64369	2.66568	3.283	
82 4-Chlorotoluene	2.58284	2.77274	2.68898	2.85256	2.80646	2.74072	3.890	
83 Ethylmethacrylate	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
84 tert-Butylbenzene	3.30636	3.34107	3.14458	3.45445	3.31883	3.31306	3.349	
85 1,2,4-Trimethylbenzene	2.53559	2.54077	2.40990	2.61253	2.53019	2.52580	2.889	
86 sec-Butylbenzene	4.37176	4.48503	4.07040	4.46310	4.36979	4.35201	3.811	
87 1,3-Dichlorobenzene	1.46990	1.52823	1.41932	1.52187	1.56628	1.50112	3.809	
88 p-Isopropyltoluene	3.57800	3.61407	3.35732	3.70783	3.57727	3.56690	3.608	
90 1,4-Dichlorobenzene	1.71907	1.66359	1.59666	1.75587	1.64970	1.67698	3.697	
91 n-Butylbenzene	3.07826	3.11441	2.90522	3.32540	3.22211	3.12908	5.056	
92 1,2-Dichlorobenzene	1.23371	1.25886	1.18402	1.27557	1.20564	1.23156	3.043	
93 1,2-Dibromo-3-Chloropropane	0.08803	0.07444	0.07551	0.07959	0.07203	0.07792	8.056	
94 1,2,4-Trichlorobenzene	0.87688	0.88559	0.85637	0.92805	0.87693	0.88476	2.993	
95 Hexachlorobutadiene	0.98300	0.99688	0.86579	1.00602	0.93746	0.95783	6.035	
96 Naphthalene	0.71663	0.76293	0.79313	0.84154	0.80137	0.78312	5.947	
97 1,2,3-Trichlorobenzene	0.65540	0.64471	0.63290	0.68947	0.64334	0.65316	3.338	
98 trans-1,4-Dichloro-2-butene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-

INITIAL CALIBRATION DATA

Start Cal Date : 04-FEB-2004 01:38
End Cal Date : 04-FEB-2004 03:21
Quant Method : ISTD
Origin : Disabled
Target Version : 3.50
Integrator : HP RTE
Method file : /var/chem/gcl3.i/020404 olcprc3w.b/olcprc3w.m
Cal Date : 20-Jul-2004 14:07 petruszj
Curve Type : Average

Compound	1.000 Level 1	2.000 Level 2	5.000 Level 3	10.000 Level 4	25.000 Level 5	$\overline{RRF}$	% RSD	
M 99 Xylene (total)	1.04726	1.19673	1.09801	1.18848	1.17145	1.14039	5.703	
100 Pentachloroethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
$ 29 1,2-Dichloroethane-d4	0.15397	0.16984	0.17768	0.16176	0.16108	0.16486	5.523	
$ 48 Toluene-d8	1.11436	1.32938	1.33813	1.19503	1.26437	1.24825	7.566	
$ 75 p-Bromofluorobenzene	0.61450	0.71557	0.71423	0.65086	0.67477	0.67398	6.392	

CONTINUING CALIBRATION COMPOUNDS

Instrument ID: gcl3.i Injection Date: 04-FEB-2004 02:29
Lab File ID: 3i0204b.d Init. Cal. Date(s): 04-FEB-2004 04-FEB-2004
Analysis Type: WATER Init. Cal. Times: 01:38 03:21
Lab Sample ID: VSTD005 Quant Type: ISTD
Method: /var/chem/gcl3.i/020404_olcprc3w_mdl.b/olcprc3w.m

COMPOUND	RRF / AMOUNT	RF5	MIN RRF	%D / %DRIFT	MAX %D / %DRIFT	CURVE TYPE
1 Dichlorodifluoromethane	0.44538	0.44606	0.010	-0.15333	40.00000	Averaged
2 Chloromethane	0.25782	0.25936	0.010	-0.59877	40.00000	Averaged
3 Vinyl chloride	0.31534	0.30804	0.100	2.31464	30.00000	Averaged
4 Bromomethane	0.20149	0.20634	0.100	-2.40779	30.00000	Averaged
8 Chloroethane	0.18780	0.18737	0.010	0.23345	40.00000	Averaged
9 Trichlorofluoromethane	0.59456	0.56024	0.010	5.77194	40.00000	Averaged
10 1,1-Dichloroethene	0.29025	0.27667	0.100	4.68184	30.00000	Averaged
11 Acetone	0.01071	0.00955	0.010	10.89986	40.00000	Averaged <- na
12 Carbon Disulfide	0.50849	0.48839	0.010	3.95099	40.00000	Averaged
14 Methylene chloride	0.25300	0.23659	0.010	6.48665	40.00000	Averaged
16 Methyl-tert-Butyl Ether	0.24713	0.24257	0.010	1.84731	40.00000	Averaged
15 trans-1,2-Dichloroethene	0.34515	0.33111	0.010	4.06910	40.00000	Averaged
18 1,1-Dichloroethane	0.61776	0.60507	0.200	2.05397	30.00000	Averaged
19 cis-1,2-Dichloroethene	0.30130	0.29867	0.010	0.87218	40.00000	Averaged
21 2-Butanone	0.01684	0.01720	0.010	-2.13766	40.00000	Averaged
20 2,2-Dichloropropane	0.48662	0.46439	0.010	4.56782	40.00000	Averaged
22 Bromochloromethane	0.14510	0.14314	0.050	1.35076	30.00000	Averaged
23 Tetrahydrofuran	0.01355	0.01309	0.010	3.40325	40.00000	Averaged
24 Chloroform	0.54389	0.53481	0.200	1.66977	30.00000	Averaged
26 1,1,1-Trichloroethane	0.77283	0.73933	0.100	4.33404	30.00000	Averaged
27 1,1-Dichloropropene	0.52580	0.48807	0.010	7.17630	40.00000	Averaged
28 Carbon tetrachloride	0.82111	0.76732	0.100	6.55053	30.00000	Averaged
$ 29 1,2-Dichloroethane-d4	0.16486	0.17768	0.010	-7.77088	40.00000	Averaged
31 1,2-Dichloroethane	0.17906	0.17702	0.100	1.14025	30.00000	Averaged
30 Benzene	1.20817	1.17492	0.400	2.75249	30.00000	Averaged
38 Trichloroethene	0.65124	0.63162	0.300	3.01167	30.00000	Averaged
40 1,2-Dichloropropane	0.44227	0.43316	0.010	2.05877	40.00000	Averaged
41 Dibromomethane	0.20830	0.20744	0.010	0.41381	40.00000	Averaged
42 Bromodichloromethane	0.59880	0.59397	0.200	0.80571	30.00000	Averaged
M 43 1,2-Dichloroethene (total)	0.32322	0.31489	0.010	2.57908	40.00000	Averaged
46 cis-1,3-Dichloropropene	0.49120	0.48474	0.200	1.31385	30.00000	Averaged
47 4-Methyl-2-pentanone	0.08482	0.08640	0.010	-1.85782	40.00000	Averaged
$ 48 Toluene-d8	1.24825	1.33813	0.010	-7.20022	40.00000	Averaged
49 Toluene	0.80041	0.78044	0.400	2.49540	30.00000	Averaged
52 trans-1,3-Dichloropropene	0.30886	0.30026	0.100	2.78513	30.00000	Averaged

CONTINUING CALIBRATION COMPOUNDS

Instrument ID: gc13.i Injection Date: 04-FEB-2004 02:29
Lab File ID: 3i0204b.d Init. Cal. Date(s): 04-FEB-2004 04-FEB-2004
Analysis Type: WATER Init. Cal. Times: 01:38 03:21
Lab Sample ID: VSTD005 Quant Type: ISTD
Method: /var/chem/gc13.i/020404_olcprc3w_mdl.b/olcprc3w.m

COMPOUND	RRF / AMOUNT	RF5	MIN RRF	%D / %DRIFT	MAX %D / %DRIFT	CURVE TYPE
54 1.1.2-Trichloroethane	0.19606	0.19303	0.100	1.54489	30.00000	Averaged
56 1.3-Dichloropropane	0.31587	0.31394	0.010	0.61074	40.00000	Averaged
55 Tetrachloroethene	0.69070	0.65383	0.100	5.33834	30.00000	Averaged
58 2-Hexanone	0.04271	0.04445	0.010	-4.07281	40.00000	Averaged
62 Dibromochloromethane	0.44486	0.42846	0.100	3.68641	30.00000	Averaged
63 1.2-Dibromoethane	0.27570	0.27368	0.100	0.73274	30.00000	Averaged
66 Chlorobenzene	0.98272	0.95680	0.500	2.63769	30.00000	Averaged
67 1.1.1.2-Tetrachloroethane	0.47758	0.47142	0.010	1.29047	40.00000	Averaged
68 Ethylbenzene	0.48266	0.46728	0.100	3.18643	30.00000	Averaged
69 p.m-Xylene	1.23498	1.18912	0.010	3.71365	40.00000	Averaged
70 o-Xylene	1.14039	1.09801	0.010	3.71626	40.00000	Averaged
71 Styrene	0.79928	0.79167	0.300	0.95301	30.00000	Averaged
73 Bromoform	0.42035	0.41941	0.050	0.22402	30.00000	Averaged
74 Isopropylbenzene	3.41972	3.26012	0.010	4.66703	40.00000	Averaged
$ 75 p-Bromofluorobenzene	0.67398	0.71423	0.200	-5.97083	30.00000	Averaged
76 1.1.2.2-Tetrachloroethane	0.22627	0.21956	0.100	2.96582	30.00000	Averaged
77 Bromobenzene	0.83979	0.83013	0.010	1.15027	40.00000	Averaged
78 1.2.3-Trichloropropane	0.10503	0.10262	0.010	2.29962	40.00000	Averaged
79 n-Propylbenzene	3.92244	3.78273	0.010	3.56186	40.00000	Averaged
80 2-Chlorotoluene	2.72025	2.59033	0.010	4.77593	40.00000	Averaged
81 1.3.5-Trimethylbenzene	2.66568	2.53459	0.010	4.91780	40.00000	Averaged
82 4-Chlorotoluene	2.74072	2.68898	0.010	1.88761	40.00000	Averaged
84 tert-Butylbenzene	3.31306	3.14458	0.010	5.08518	40.00000	Averaged
85 1.2.4-Trimethylbenzene	2.52580	2.40990	0.010	4.58828	40.00000	Averaged
86 sec-Butylbenzene	4.35201	4.07040	0.010	6.47096	40.00000	Averaged
87 1.3-Dichlorobenzene	1.50112	1.41932	0.400	5.44947	30.00000	Averaged
88 p-Isopropyltoluene	3.56690	3.35732	0.010	5.87570	40.00000	Averaged
90 1.4-Dichlorobenzene	1.67698	1.59666	0.400	4.78959	30.00000	Averaged
92 1.2-Dichlorobenzene	1.23156	1.18402	0.400	3.86016	30.00000	Averaged
91 n-Butylbenzene	3.12908	2.90522	0.010	7.15406	40.00000	Averaged
93 1.2-Dibromo-3-Chloropropane	0.07792	0.07551	0.010	3.09548	40.00000	Averaged
94 1.2.4-Trichlorobenzene	0.88476	0.85637	0.010	3.20931	40.00000	Averaged
95 Hexachlorobutadiene	0.95783	0.86579	0.010	9.60918	40.00000	Averaged
96 Naphthalene	0.78312	0.79313	0.010	-1.27780	40.00000	Averaged
97 1.2.3-Trichlorobenzene	0.65316	0.63290	0.010	3.10229	40.00000	Averaged
M 99 Xylene (total)	1.14039	1.09801	0.300	3.71626	30.00000	Averaged

Table 1. Contract Required Quantitation Limits (CRQL)[1]

Compound	CAS Number	CRQL (ug/L)
Chloromethane	74-87-3	1
Bromomethane	74-83-9	1
Vinyl Chloride	75-01-4	1
Chloroethane	75-00-3	1
Methylene Chloride	75-09-2	1
Acetone	67-64-1	5
Carbon Disulfide	75-15-0	1
1,1-Dichloroethene	75-35-4	1
cis-1,2-Dichloroethene	156-59-2	1
trans-1,2-Dichloroethene	156-60-5	1
1,2-Dibromo-3-chloropropane	96-12-8	1
1,2-Dibromoethane	106-93-4	1
1,2-Dichlorobenzene	95-50-1	1
1,3-Dichlorobenzene	541-73-1	1
1,4-Dichlorobenzene	106-46-7	1
1,1-Dichloroethane	75-35-3	1
Chloroform	67-66-3	1
1,2-Dichloroethane	107-06-2	1
2-Butanone	78-93-3	5
1,1,1-Trichloroethane	71-55-6	1
Carbon Tetrachloride	56-23-5	1
Bromodichloromethane	75-27-4	1
1,1,2,2-Tetrachloroethane	79-34-5	1
1,2-Dichloropropane	78-87-5	1
trans-1,3-Dichloropropene	10061-02-6	1
Trichloroethene	79-01-6	1
Dibromochloromethane	124-48-1	1
1,1,2-Trichloroethane	79-00-5	1
Benzene	71-43-2	1
cis-1,3-Dichloropropene	10061-01-5	1
Bromoform	75-25-2	1
2-Hexanone	591-78-6	5
4-Methyl-2-pentanone	108-10-1	5
1,2,4-Trichlorobenzene	120-88-1	1
Tetrachloroethene	127-18-4	1
Toluene	108-88-3	1
Chlorobenzene	108-90-7	1
Ethyl benzene	100-41-4	1
Styrene	100-42-5	1
Total Xylenes		1

[1]Sample CRQLs are highly matrix-dependent. The CRQLs listed herein are provided for guidance and may not always be achievable. See the following information for further guidance on matrix-dependent CRQLs.

Table 2. Retention Times and Characteristic Ions for Volatile Compounds

Compound	Retention Time (min)	Primary Quant Ion	Secondary Ions
Acetone	5.7	43	58
Benzene	8.9	78	52, 77
Bromochloromethane	8.2	128	49, 130, 51
Bromodichloromethane	10.2	83	85, 129
4-Bromofluorobenzene (surr.)	13.9	95	174, 176
Bromoform	13.6	173	171, 175, 252
Bromomethane	4.3	94	96, 79
2-Butanone	7.9	43	57, 72
Carbon disulfide	6.0	76	78
Carbon Tetrachloride	8.7	117	119, 121
Chlorobenzene	12.6	112	114, 77
Chlorobenzene-d_5 (I.S.)	12.6	117	82, 119
Chlorodibromomethane	11.9	129	208, 206
Chloroethane	4.4	64	66, 49
Chloroform	8.3	83	85, 47
Chloromethane	3.6	50	52, 49
1,2-Dibromo-3-chloropropane	16.4	75	155,157
1,2-Dibromoethane	12.1	107	109,188
1,2-Dichlorobenzene	15.5	146	148,150
1,3-Dichlorobenzene	15.1	146	148,150
1,4-Dichlorobenzene	15.2	146	148,150
1,1-Dichloroethane	7.2	63	65, 83
1,2-Dichloroethane	8.9	62	64, 98
1,1-Dichloroethene	5.6	96	61, 98
cis-1,2-Dichloroethene	7.9	96	61, 63
trans-1,2-Dichloroethene	6.7	96	61,98
1,2-Dichloropropane	9.9	63	62, 41
cis-1,3-Dichloropropene	10.7	75	77, 39
trans-1,3-Dichloropropene	11.3	75	77, 39
1,4-Difluorobenzene (I.S.)	9.3	114	63, 88
Ethylbenzene	12.7	106	91
2-Hexanone	11.6	43	58, 57, 100
Methylene chloride	6.3	84	49, 51, 86
4-Methyl-2-pentanone	10.8	43	58, 100
Styrene	13.4	104	78, 103
1,1,2,2-Tetrachloroethane	14.0	83	85, 131, 133
Tetrachloroethene	11.7	166	129, 131, 168

Table 2. Retention Times and Characteristic Ions for Volatile Compounds (continued)

Compound	Retention Time (min)	Primary Quant Ion	Secondary Ion
Toluene	11.1	91	92, 65
1,2,4-Trichlorobenzene	14.8	105	182,145
1,1,1-Trichloroethane	8.5	97	99, 117
1,1,2-Trichloroethane	11.5	97	83, 85, 99
Trichloroethene	9.6	130	95, 97, 132
Vinyl chloride	3.7	62	64, 61
1,2-Dichlorobenzene-d4	15.2	152	150,115
Xylene (total)	---	91	106

NOTE: The primary and secondary ions listed may differ slightly from OLC02.1 but are consistent across analytical methods performed at this laboratory.

Retention times are generated from a specific system.

Xylene is quantitated against mass 91. It is used to help the analysts differentiate between ethylbenzene and xylene.

Attachment 1.

Examples: ID File Listing; **Target Compounds and Internal Standards;** Initial/Continuing Calibration Requirements; **Initial Calibration/Continuing Calibration;** Surrogate Recovery Limits; **LCS / MS Spike Recovery Limits**

Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

1,4-Difluorobenzene
Acetone
Bromochloromethane
Bromomethane
2-Butanone
Carbon disulfide
Chloroethane
Chloroform
Chloromethane
1,1-Dichloroethane
1,2-Dichloroethane
1,1-Dichloroethene
cis-1,2-Dichloroethene
trans-1,2-Dichloroethene
Methylene chloride
Vinyl chloride

Chlorobenzene-d_5
Benzene
Bromodichloromethane
Chlorobenzene
Carbon tetrachloride
Chlorodibromomethane
1,2-Dibromoethane
1,2-Dichloropropane
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Ethylbenzene
2-Hexanone
4-Methyl-2-pentanone
Styrene
1,1,2,2-Tetrachloroethane
Tetrachloroethene
Toluene
Xylenes(total)
Trichloroethene
4-Bromofluorobenzene

1,4-Dichlorobenzene-d_4
Bromoform
1,2-Dibromo-3-chloropropane
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2,4-Trichlorobenzene

Initial/Continuing Calibration Criteria

The response factors of the compounds listed below must meet the minimum RRF criteria at each concentration level and maximum %RSD criteria for the initial calibration, with allowance made for up to two volatile compounds. However, the RRFs for those two compounds must be greater than or equal to 0.010, and the %RSD of those two compounds must be less than or equal to 40.0% for the initial calibration to be acceptable.

Volatile Compound	Minimum RRF	Maximum %RSD	Maximum %Diff.
Bromomethane	0.100	30.0	30.0
Vinyl chloride	0.100	30.0	30.0
1,1-Dichloroethene	0.100	30.0	30.0
1,1-Dichloroethane	0.200	30.0	30.0
Chloroform	0.200	30.0	30.0
1,2-Dichloroethane	0.100	30.0	30.0
1,1,1-Trichloroethane	0.100	30.0	30.0
Carbon tetrachloride	0.100	30.0	30.0
Bromodichloromethane	0.200	30.0	30.0
cis-1,3-Dichloropropene	0.200	30.0	30.0
Trichloroethene	0.300	30.0	30.0
Dibromochloromethane	0.100	30.0	30.0
1,1,2-Trichloroethane	0.100	30.0	30.0
Benzene	0.400	30.0	30.0
trans-1,3-Dichloropropene	0.100	30.0	30.0
Bromoform	0.050	30.0	30.0
Tetrachloroethene	0.100	30.0	30.0
1,1,2,2-Tetrachloroethane	0.100	30.0	30.0
Toluene	0.400	30.0	30.0
Chlorobenzene	0.500	30.0	30.0
Ethylbenzene	0.100	30.0	30.0
Styrene	0.300	30.0	30.0
Xylenes (total)	0.300	30.0	30.0
Bromofluorobenzene	0.200	30.0	30.0
1,2-Dibromoethane	0.100	30.0	30.0
1,2-Dichlorobenzene	0.400	30.0	30.0
1,3-Dichlorobenzene	0.400	30.0	30.0
1,4-Dichlorobenzene	0.400	30.0	30.0

The following compounds have no Maximum %RSD, or Maximum %Difference criteria; however, these compounds must meet a minimum RRF criterion of 0.010:

1,2-Dichloropropane
Carbon disulfide
Chloroethane
Chloromethane
cis-Dichloroethene (total)
trans-Dichloroethene
cis-Dichloroethene
Methylene chloride

System Monitoring Compound Recovery Limits

Compound	% Recovery (Water)
Bromofluorobenzene	80 – 120

LCS / MS Recovery and Relative Percent Difference Limits

Compound	% Recovery (Water)	RPD (Water)
1,2-Dibromoethane	60 - 140	40
1,4-Dichlorobenzene	60 - 140	40
Trichloroethene	60 - 140	40
cis-1,3-Dichloropropene	60 - 140	40
Benzene	60 - 140	40
1,2-Dichloroethane	60 - 140	40
1,1,2-Trichloroethane	60 - 140	40
1,2-Dichloropropane	60 - 140	40
Vinyl Chloride	60 - 140	40
Bromoform	60 - 140	40
Carbon Tetrachloride	60 - 140	40
Tetrachloroethane	60- 140	40

Internal Standard Limits

Internal standard areas will not deviate by -/+40% of the current continuing calibration. Retention times will not deviate by $\pm$0.33 minutes.

Attachment 2.

Example: Method Listings; Concentrator and Archon Conditions; **Flow Settings**

Example: Volatiles Method for 5971/5972

GC Oven Parameters

Initial Temperature = 40 °C
Initial Time = 2.0 minutes
Detector A Temperature = 180 °C
Detector B Temperature = 250 °C
Oven Equib. Time = 0.50 min.

Ramp Rate (°C/min.)	Final Temp. (°C)	Final Time (min.)
7.0	65	0.00
12.0	165	0.00
20.0	212	5.00

Run Time = 21.25 min.

Inlet Pressure Program

Gas = Helium
Column length = 75 m
Column Diameter = 0.530 mm
Initial Pressure = 3 psi
Rate (psi/min) = 0.00
Initial Time = 7.0 min.
Oven Temp. 50 °C
Program Time = 7.0 min.

Scan Parameters

Mass Range = 35-260
Threshold = 150
Scans/sec = 1.9
EM Voltage = 1938
Solvent Delay (scan start time): before the elution of the first compound.
Run Time (scan stop time): until after the elution of last compound.

Example: Volatiles Method for 5973

GC Oven Parameters

Initial Temperature = 50 °C
Initial Time = 2.0 minutes
Aux Temperature = 250 °C
Oven Equib. Time = 0.50 min.

Ramp Rate (°C/min.)	Final Temp. (°C)	Final Time (min.)
15.0	220	0.00

Run Time = 13.33 min.

Inlet Pressure Program

Mode=split
Gas = Helium
Column length = 25 m
Column Diameter = 0.25 mm
Constant flow = 1.0 mL/min
Injection port temp. = 250 °C
Program Time = 7.0 min.
Split ratio = 80:1
Gas saver = off

Scan Parameters

Mass Range = 35-260
Threshold = 100
Scans/sec = 6
EM Voltage = 1938
Solvent Delay = 0.8 min. (scan start time): before the elution of the first compound.
Run Time (scan stop time): until after the elution of last compound.

Concentrator Conditions

Trap Temp. Prior to Purge	< 35
Desorb Preheat	250
Desorb	250
Bake	260
Purge Time	11 min
Desorb	0.5-2 min (inst. dependent)
Bake Time	4-6 min

Trap = Vocarb 3000

Flow Conditions

Purge Pressure	20 psi
Purge Flow Rate	~40 mLs/min

Flow Adjustment

Capillary Column: 5971/5972/MSD's;

- Make-up gas off/separator pump on: flow through separator is 5-10 mLs/minutes.
- Open make-up gas: adjust until you achieve ~30 mLs/minute through the separator. (On MSD's - adjust to 0.5 torr on gauge)

(Flow into the Mass Spec is $\leq$ 1 mL/minute)

Approximate Vacuums

~5 x 10^{-6} torr

Example Archon Conditions

Transfer Line Temp	110 deg C
Soil Valve	95 deg C
Purge Pressure	25 psi
Purge Flow	~ 40 ml/min
Purge Time	11 min
Desorb	0.5 -2 min
SamplePre-heat(soils)	40 deg C ~ 2 min

Attachment 3.

Examples: Analysis / CAR-Qualification Logbook; Maintenance Logbook; Tune Form; and Sample Tracking Sheet

STL Chicago
GC/MS Volatile Analysis Logbook

Instrument ID# 17

CHI-22-20-054/B-12/03

Analysis Date / Time	File Name	Sample Number	Sample ID	Sparge No.	Sample Wt. / Vol.	Instr. Dil.	Int. Std. No.	pH < 2	Comments (MUST include SRN's)	Analyst Initials

Analyst Signature/Date:__________ Reviewer Signature/Date:__________ Page No.______

Instrument ID# 17

STL Chicago

Corrective Action/Qualification Report GC/MS VOA

CHI-22-20-054/B-12/03

Analytical Methods
___SW846 8260
___40CFR 624
___OLM04.2

Tune Name:____________'04
___EPA 524.2 __Other____________
___OLM03.2
___OLC02.1

ISTD/RT report
Initial calibration / Continuing Calibration IS#______________
(For method 524 include all points in ICAL)

Data File Name:

	IS 1	RT1	IS2	RT2	IS3	RT3	IS4	RT4
1								
2								
3								
4								

Tune Criteria
Description of Situation:____________
Action Taken:____________
Demonstration of Control:____________

Initial Calibration Cirteria
Description of Situation:____________
Action Taken:____________
Demonstration of Control____________

Continuing Calibration Criteria
Description of Situation:____________
Action Taken:____________
Demonstration of Control:____________

Internal Standards (continuing cal to continuing cal)
Description of situation:____________
Action Taken:____________
Demonstration of Control:____________

Method Blank
Description of situation:____________
Action Taken:____________
Demonstration of Control:____________

LCS
Description of Situation:____________
Action Taken:____________
Demonstration of Control:____________

Qualification of Data
Data Affected (Client/Sample #)____________
Qualification:____________

Associated samples reanalyzed: Yes No (see below)
Explanation for no reanalysis/data MUST be qualified and narrated:__________

Analyst Signature/date____________/__________

Reviewer Signature/date____________/__________

Date of Maintenance:__________ Entry No.:__________

Analyst:__________

Description:__________

Follow-Up:__________

Analyst:__________ Date:__________

Date of Maintenance:__________ Entry No.:__________

Analyst:__________

Description:__________

Follow-Up:__________

Analyst:__________ Date:__________

Date of Maintenance:__________ Entry No.:__________

Analyst:__________

Description:__________

Follow-Up:__________

Analyst:__________ Date:__________

Reviewer Signature or Initials / Date:__________

Tune Name: ____ CV File: ____ STD Code: ____ Method: ____ Tune Batch: ____
Tune Time: ____ CV File: ____ STD Code: ____ Method: ____ Tune Batch: ____
INST #: LCS: Method: Tune Batch:

Comments:

Sample ID:	File:	Act		pH	TS	SS	Misc. Info	Prep Method	Prep Batch	Analytical Batch

WITHIN TUNE TIME ☐

rpjah

Job Analysis History V2

06/28/2004

Surrogate Reagent...:

Method Code....: 503025 5030 25 mL Purge Prep
Job Number.....: 228041 Customer Job ID..: KI SAWYER
Project Number.: 20002287 KI Sawyer

Holding Time.....: 14 Day Holding Time
Customer.........: White Water Associates, Inc.
Proj. Cat..: USACE PM...: rcw Hardcopy Due Date.:

Job Report Type...: l4qfafce
Contact.: Bette Premo
Fax Due Date.:

Sample #	QC	Client Sample ID	Matrix	HT Date	TAT Date	File Name	Dil	Tune name	Action	Analst	Prep Batch	Comments
6	N	1004GML7MN	WATER	07/09/2004	07/16/2004							
7	N	1004GML7DN	WATER	07/09/2004	07/16/2004							
8	N	1004GML6SB	WATER	07/09/2004	07/16/2004							
9	N	1004GML6SF	WATER	07/09/2004	07/16/2004							
10	Y	1004GML6SN	WATER	07/09/2004	07/16/2004							

Attachment 4.

Example: Data Review Checklist

STL Chicago
GCMS DATA REVIEW CHECKLIST

Site Name:________________________ Primary Reviewer:_________________________ Review Date:_____________
JOB Number: _______________________ Secondary Reviewer:_______________________ Review Date:_____________
No. of Samples/Matrix: a) ______ WATER b) ______ SOIL c) ______ SPLP / TCLP d) ______ Other (________________)
Method: a) VOA ____________ 5030______ Encores: 5035–High_______ 5035-Low______ b) BNA _______________
Report Type: a) MDL U b) RL U c) ND d) Breach e) P1 / P2 (Printed QC must match PM selected Report Type)

TASK	PRI REV	SEC REV	COMMENTS
LAB CHRON: 1) Matches Big Board (Job Analysis History)			
2) Matches Raw Data (Form 4 / 5)			
3) Note Sample dilutions and list reason. a) High Sample Conc. b) Interference present IF original and re-run are to be reported in LabNet Re-log Samples (Indicate data type used) Re-Analyzed (RE) Re-Extracted (RA) Dilution (DL)			Smp # Original Dilution Comments BNA Only: Final Volume Adjustment __________
4) Sample Hold Times Met			
5) Proper Prep Links Created S-F6: Routine Preps; 5035PL; 5035PH S-F9: TCLP; SPLP ; 5035 Archon Purge & Trap			
Incomplete JOB Status Report reveals No Outstanding Data			
PROJ. REQ. MET: 1) Sample Detection Limit Met			
2) Reported J values meet reporting criteria			
3) Method Blank Detection Limits Met			
4) Project Specific Compounds of Concern : Yes No			
LabNet Report matches Quant Report: Sample weights / Volumes / % Moisture / Factors verified			
Manual Integration documentation / verification complete			
FORM 2: Surrogate Recoveries Within Limits Statistical Limits _____ Method Limits _____ Project Limits _____(S-F10 used to Clone By Project) AFCEE___; LCG___; QAPP___ Directed Note to PM: Yes / No			Smp # Original Re-analysis Comments
FORM 3: MS/MSD Recoveries Acceptable Statistical Limits _____ Method Limits _____ Project Limits _____(S-F10 used to Clone By Project) AFCEE___; LCG___; QAPP___ Directed Note to PM: Yes / No			Smp # ______ MS MSD RPD
FORM 3: LCS Recoveries Acceptable (LCD if no MS/MSD) Statistical Limits _____ Method Limits _____ Project Limits _____(S-F10 used to Clone By Project) AFCEE___; LCG___; QAPP___ Directed Note to PM: Yes / No			Batch # __________ Batch # __________ Batch # __________ Batch # __________ Batch # __________ Batch # __________

TASK	PRI REV	SEC REV	COMMENTS
FORM 5: Tuning Criteria Met			
FORM 6: Initial Calibration Criteria Met			
ICAL Spike Required: Yes No Control Limit applied: ______________			
FORM 7: Daily Calibration (CCV) Criteria Met			
MRL Check Required: Yes No (LCG Requirement – Before and after Sample analysis) Control Limit Applied: ______________			Before: After: Batch # ________ Batch # ________ Batch # ________ Batch # ________ Batch # ________
FORM 8: Internal Standards Criteria Met Directed Note to PM: Yes / No			Smp # Original Re-analysis Comments
LabNet Batch Status Report Displays Data at RPT / RVWD Status	RPT	RVWD	
RAW DATA: 1) Raw Data Verified/Complete			
2) Raw Data Matches Forms			
3) 5035 Prep Log page present / verified			
4) Quant Report Matches Spectra			
5) Manual integration reports (befores and afters) present (when required by client) and reason correctly documented and approved. Manual Integration Summary Printed: Yes No			File ID:
NARRATIVE: 1) Holding Times			
2) Method References			
3) % Recoveries / RPD's			
4) Analytical Difficulties/Typos/CAR's Directed Note to PM: Yes / No			
Manual Calculation of On Column result: Response Factor (Smp) x Concentration of IS IS Response Factor (Smp) Cmpd. RRF (Cont.Calib)			Sample : __________ Compound: ______________

Additional Comments:

CHI-22-20-038/L-12/02

Attachment 5.

Analysis and Sample Tracking Flowcharts

ANALYSIS SCHEME FLOWCHART
(Terms defined in the Section 9)

```
                    PFTBA
                      |
                      |
                      |
                     BFB  (Form 5)
                     / \ Every 12 hours
                    /   \
                   /     \
      Initial Calibration--------Continuing Calibration
         (Form 6)                     (Form 7)
                                  Every 12 hours
                                        |
                                        |
                                       \|/
                                      Analysis
                                        |
                                        |
                                       \|/
                           Computer Data Production/Reduction
                                        |
                                        |
                                       \|/
                            Operator Data Review/Reduction
                                  (Form 1,2,3,4,5)
                                        |
                                        |
                                       \|/
                             Final Review/Data Submission
                                        |
                                        |
                                       \|/
                                 Report Generation
```

Sample Tracking Flowchart (for EACH unique Sample Batch #)

```
                    Sample Receipt
                         | \
                         |  \
                      Log-In   VOA Refrigerator
                      /  |      |
                     /   |      |
                    /    |      |
                 COC  Computer  |
                  |      |      |
                  |      |      |
                  |      |      |
                  |   Backlog   |
                  |      |      |
                  |\     |     /
                  | \    |    /
                  |  \   |   /
                  |   \  |  /
                 VOAs   VOAs
                  |      |
                  |      |
                 Filed  Posted
                   \    /
                    \  /
                     \/
                    Input
                      |
             Sample Tracking Board
                      |
                  Analysis
                      |
OUTPUT:COC, Tune Form, Oper. Anal. Logbook, Comp.
        Data Prod., Tune, Calibration, QC
             _________|_________
· Operator Review/Data Reduction        ·Archival (Automated scheduled back-ups)
· Data Package w/ Review Form, CARs     · Tune Form filed
· 1st 100% Review                       · Review Form, CAR copies filed
· Submission/Final Review               · Data purged off system
      |_________________________________________________|
                          |
                  REPORT GENERATION
```